

EL889192337US

TO2017-5296500T

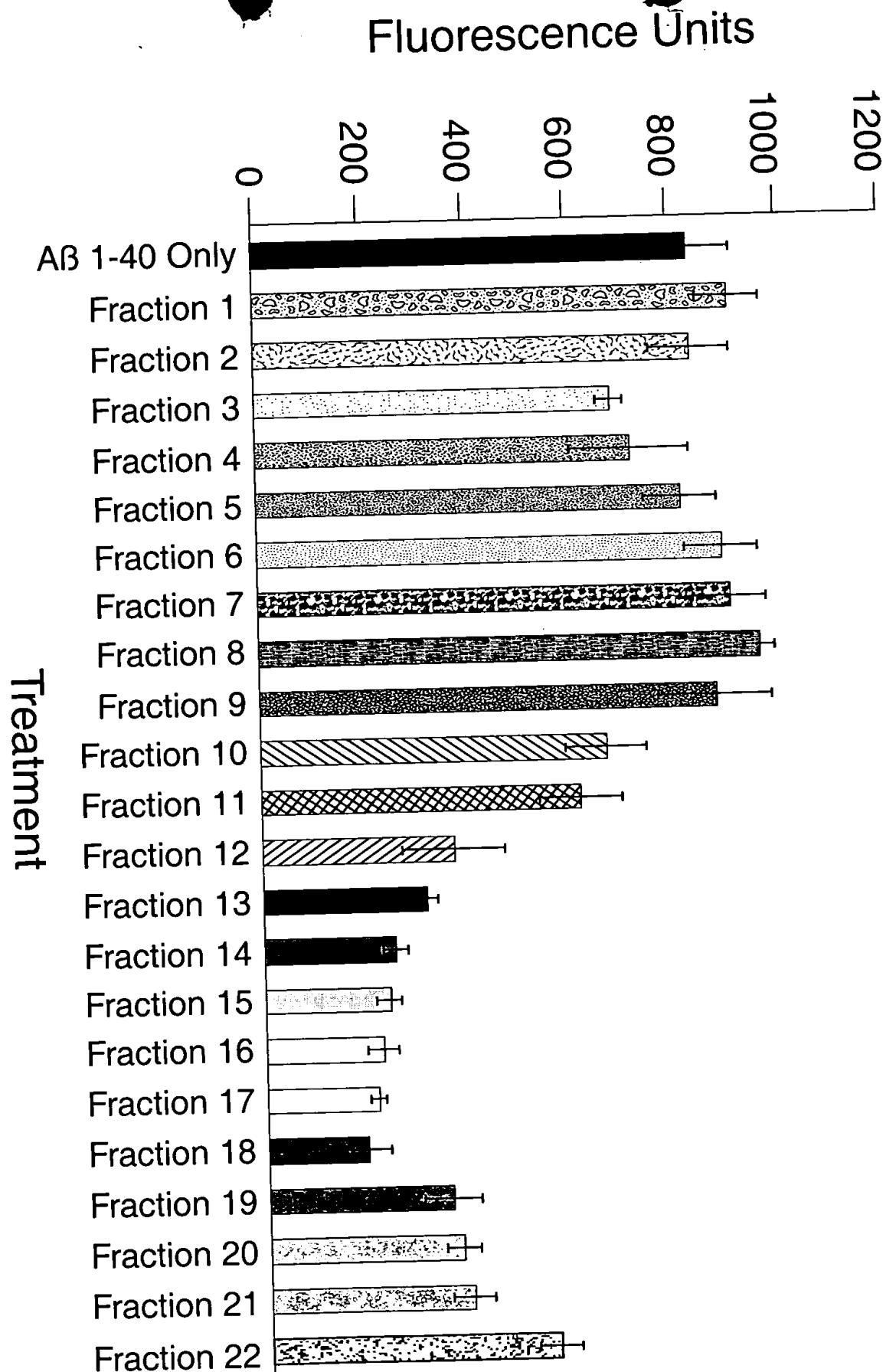


FIGURE 1

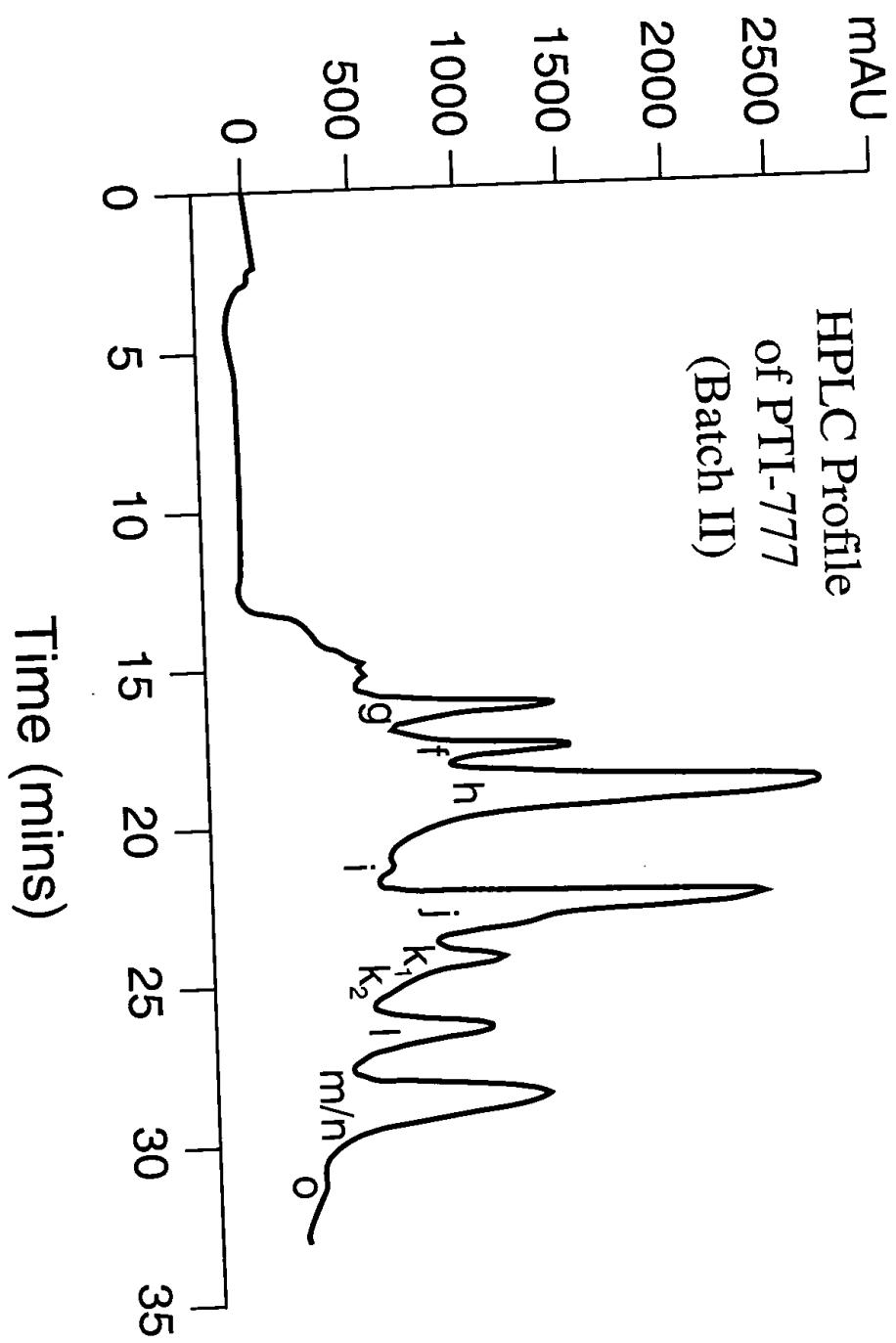
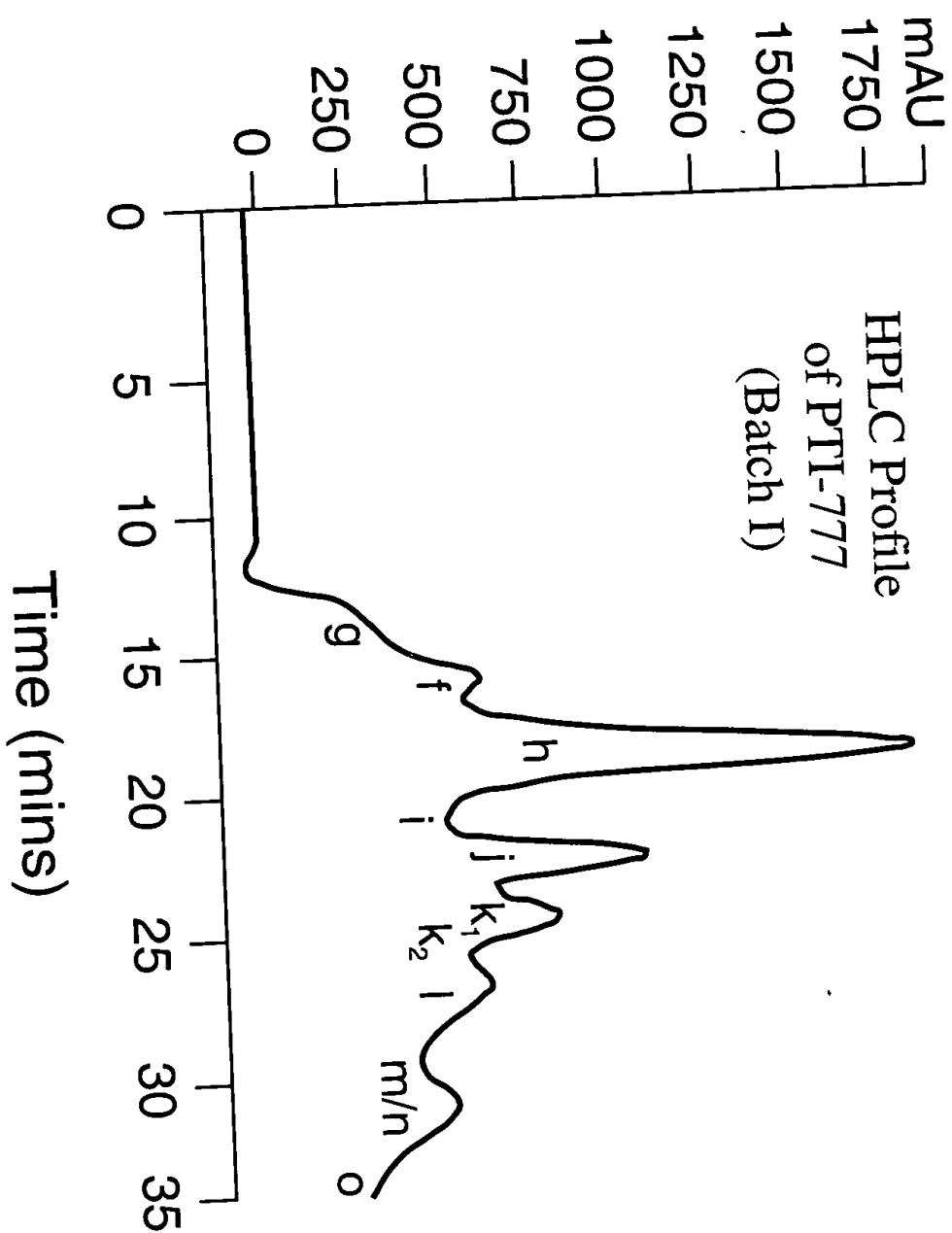
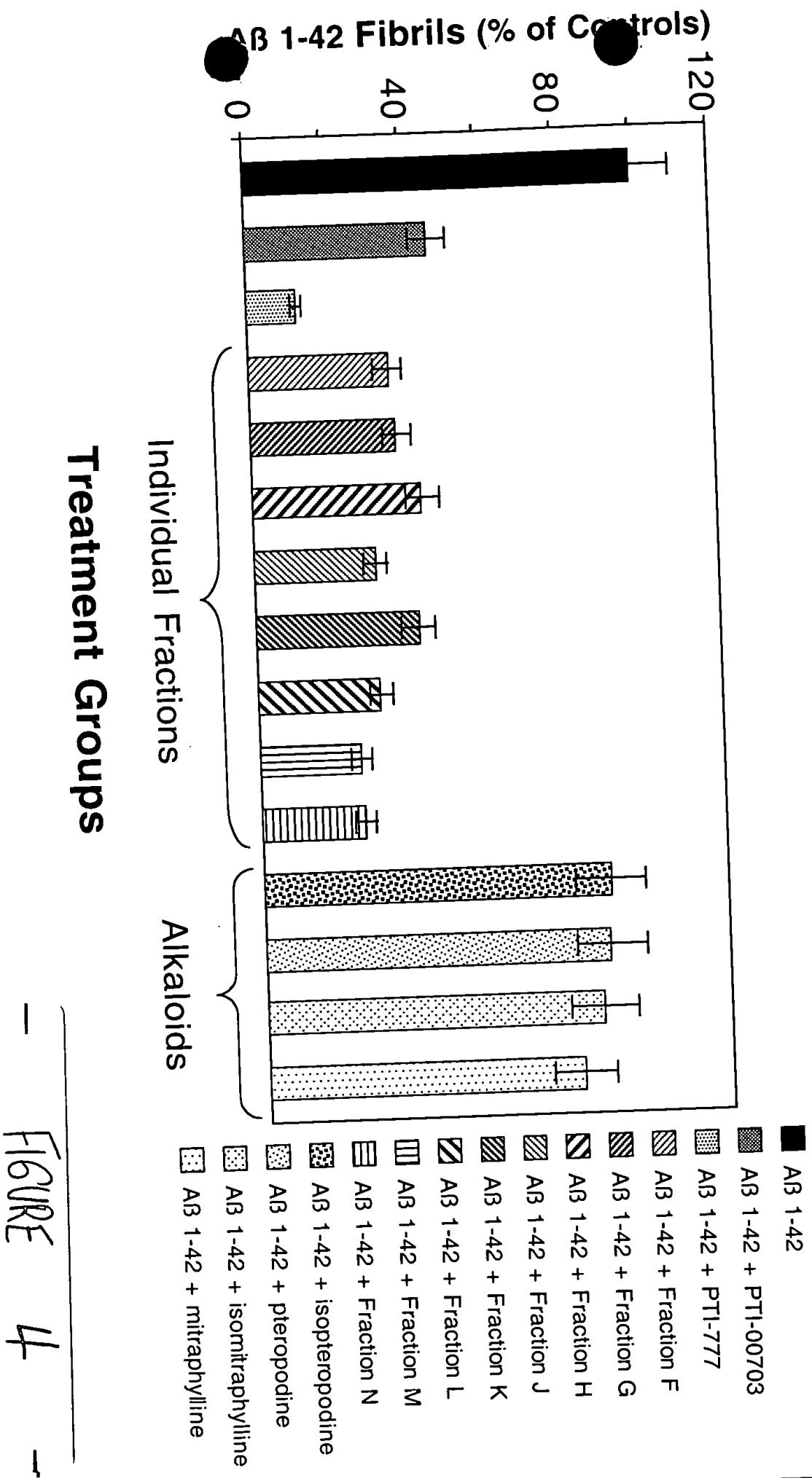


FIGURE 2



— FIGURE 3 —



THOMAS GAGEWOOD



— FIGURE 5 —

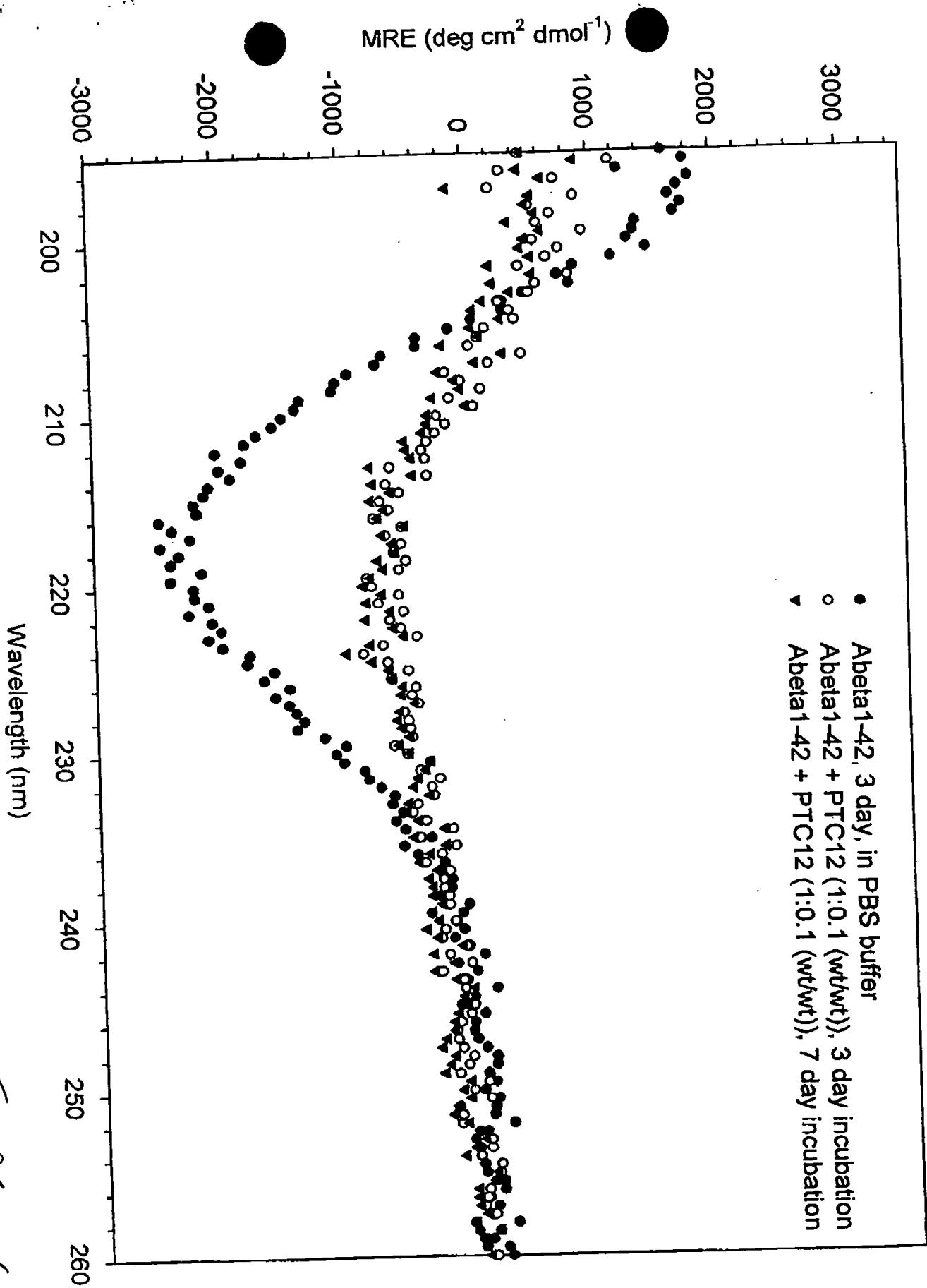


FIGURE 6

TOPOTYPE - SAMPLING SITE

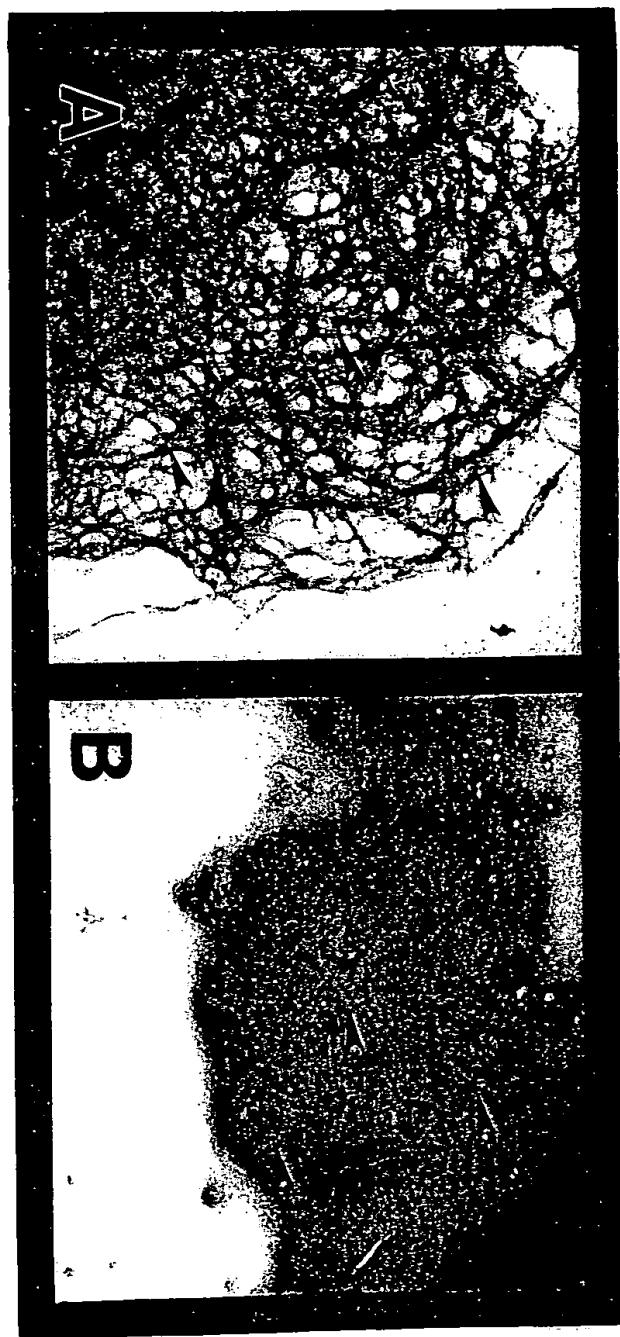
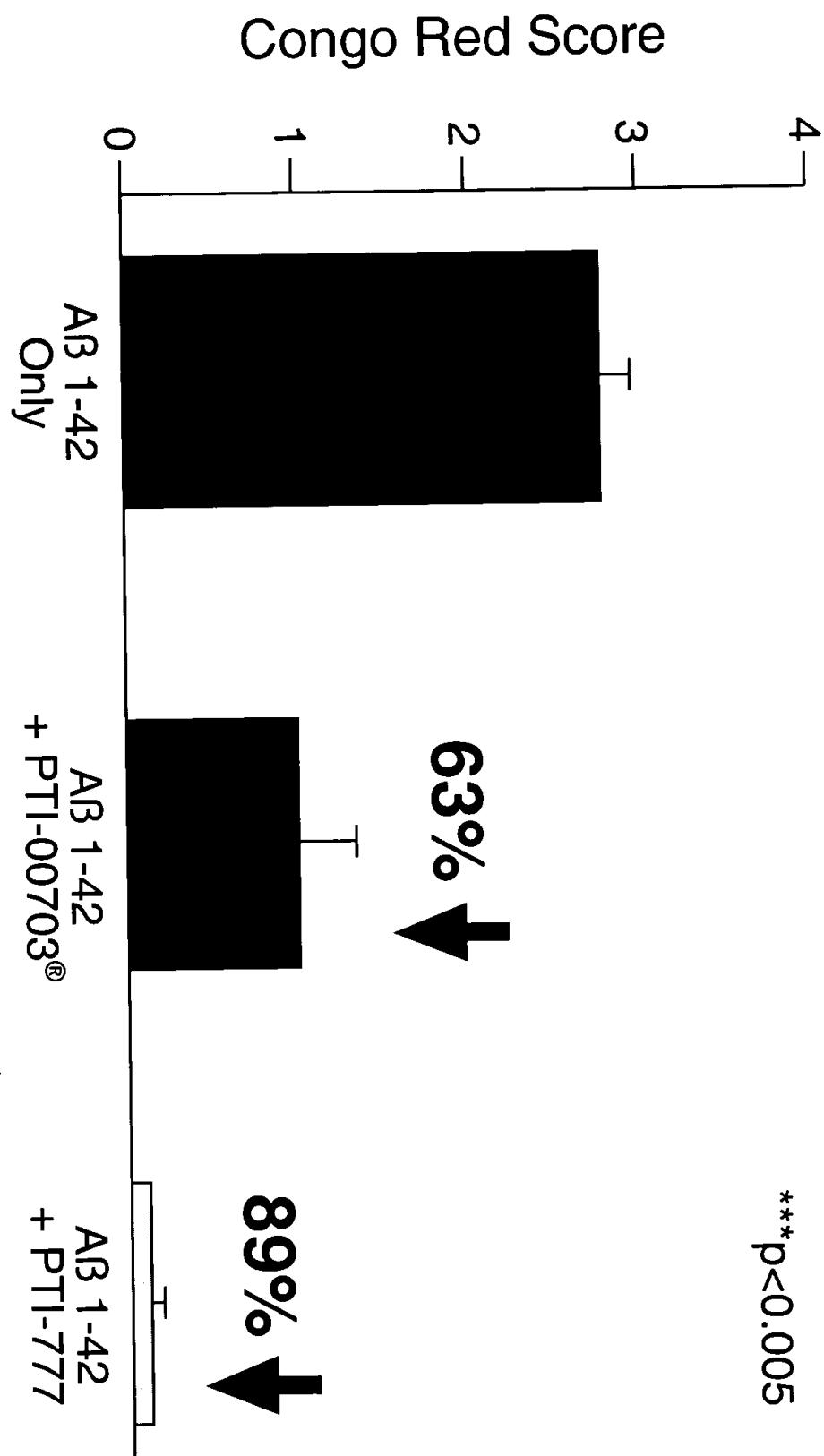


FIGURE 7



TO 20 TT - 529500

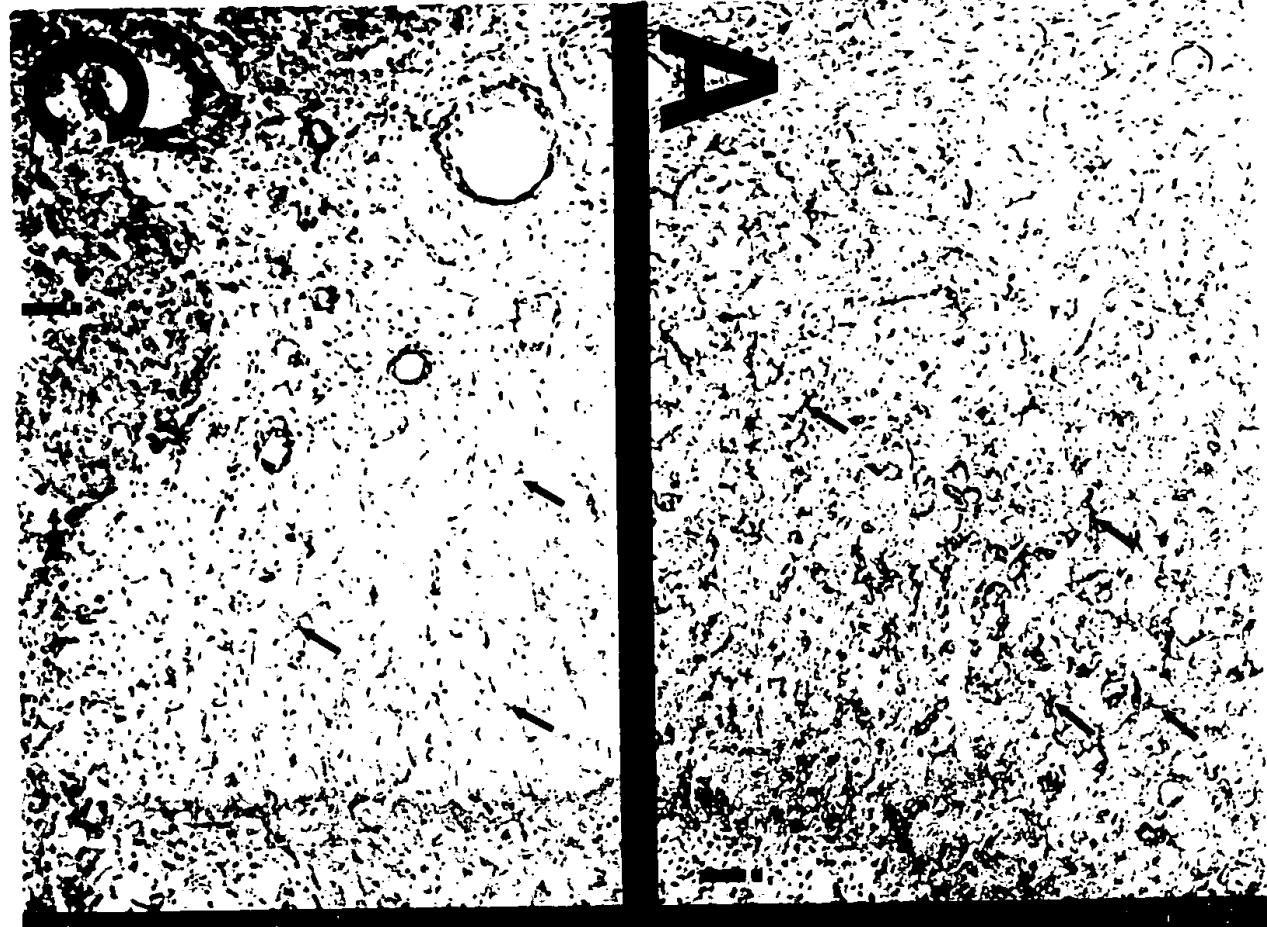
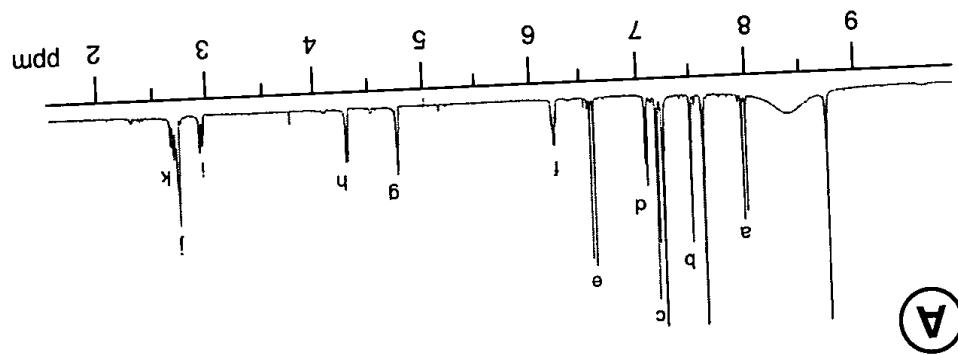
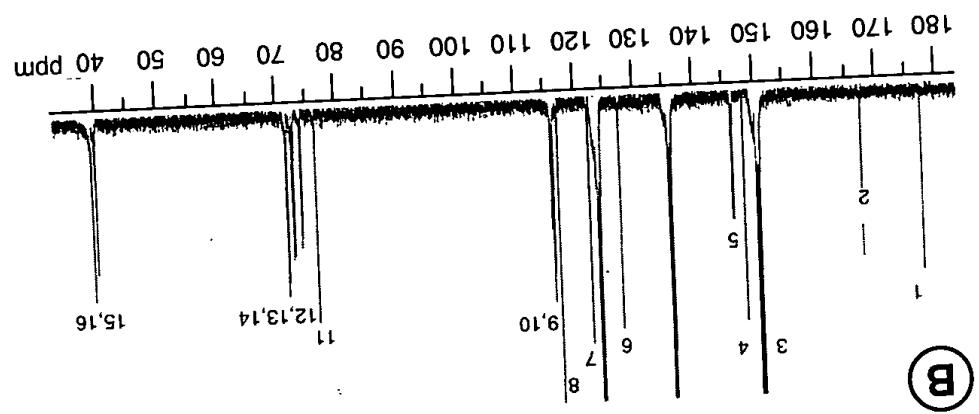
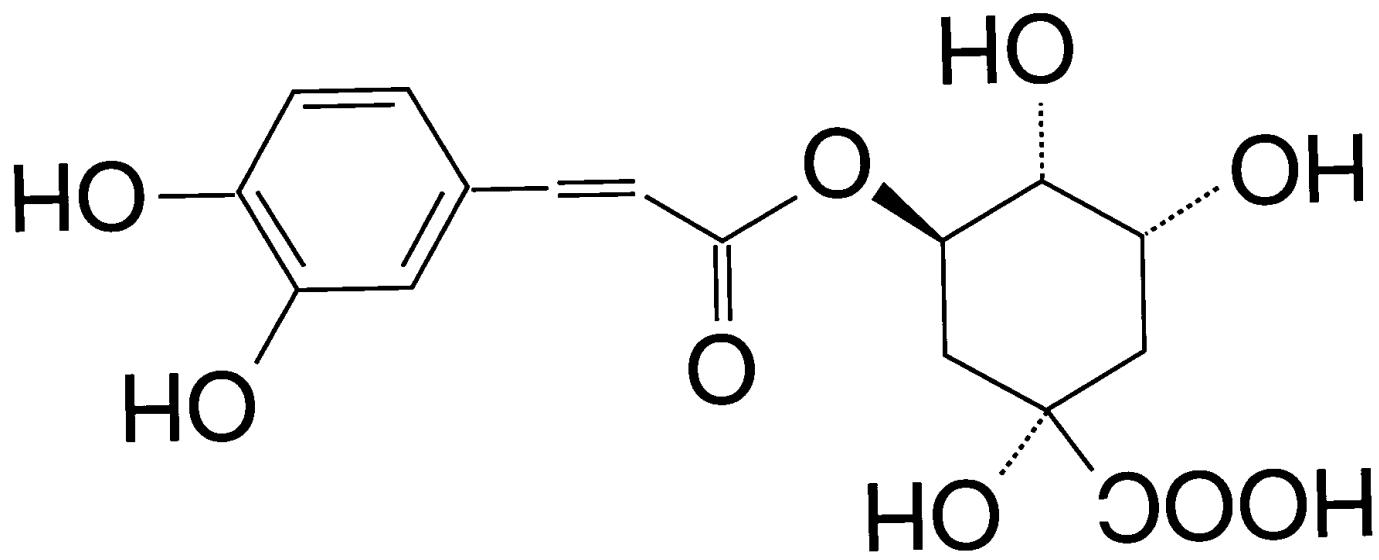


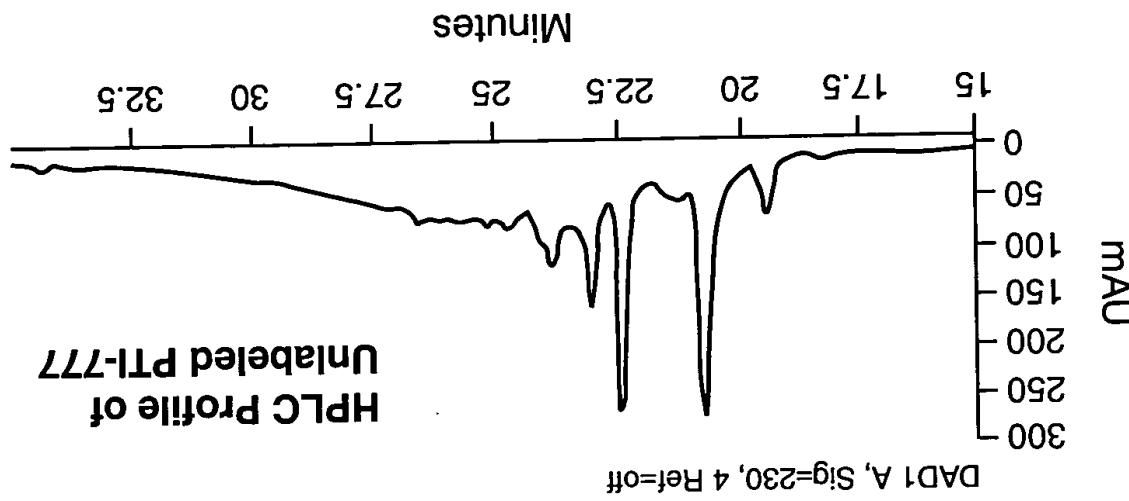
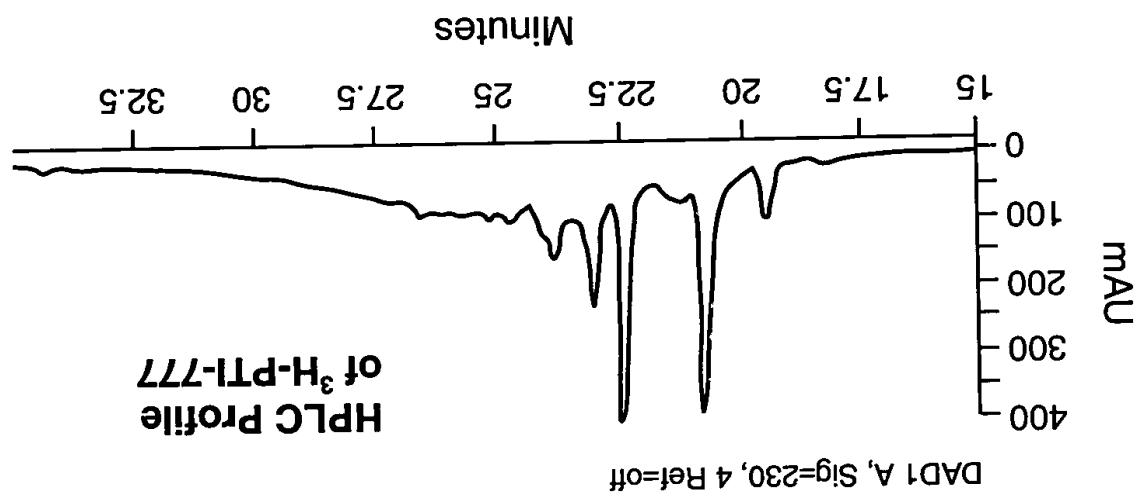
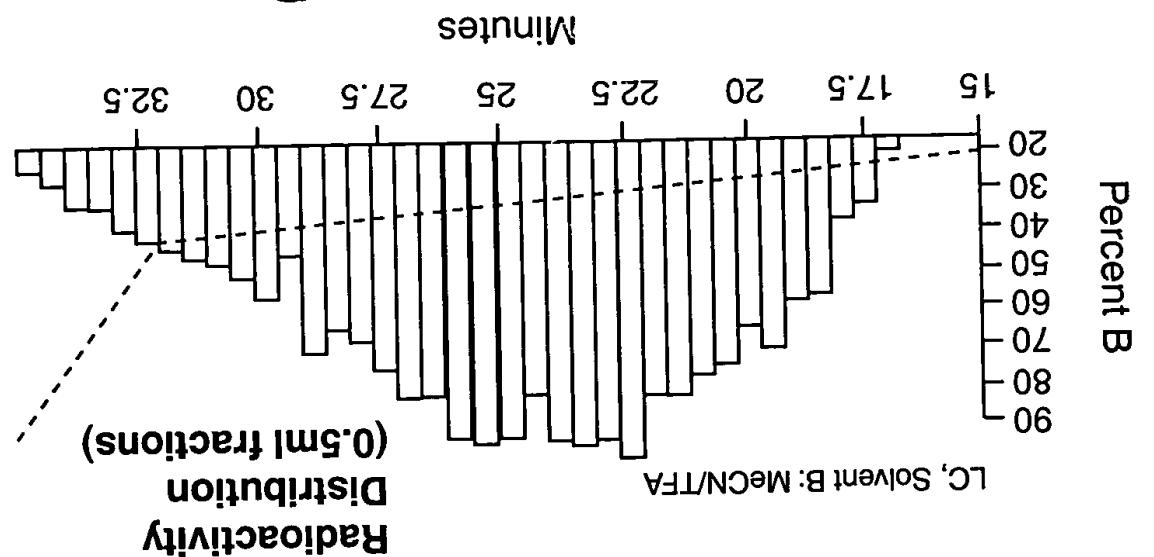
FIGURE 10

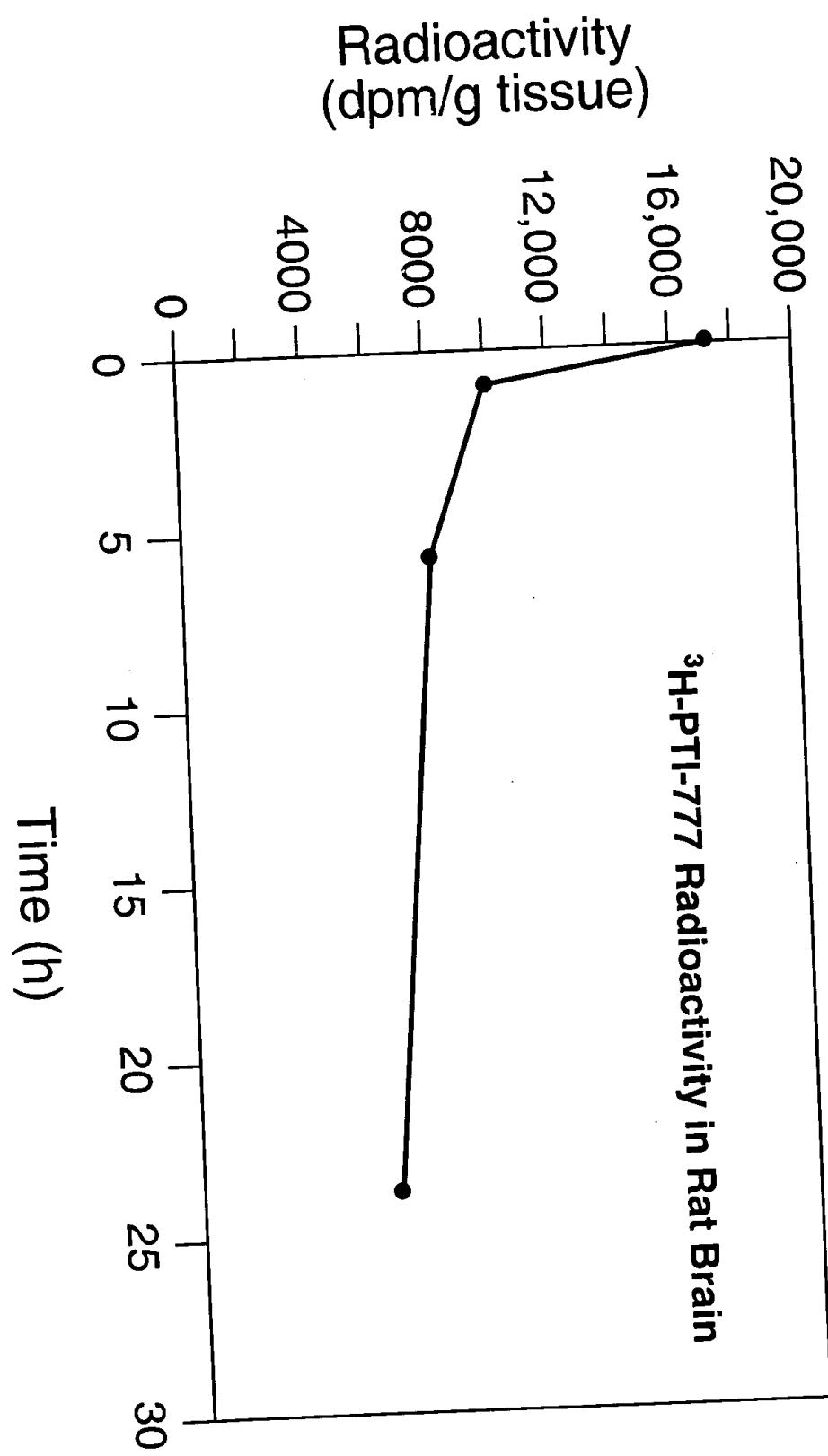
FIGURE 10



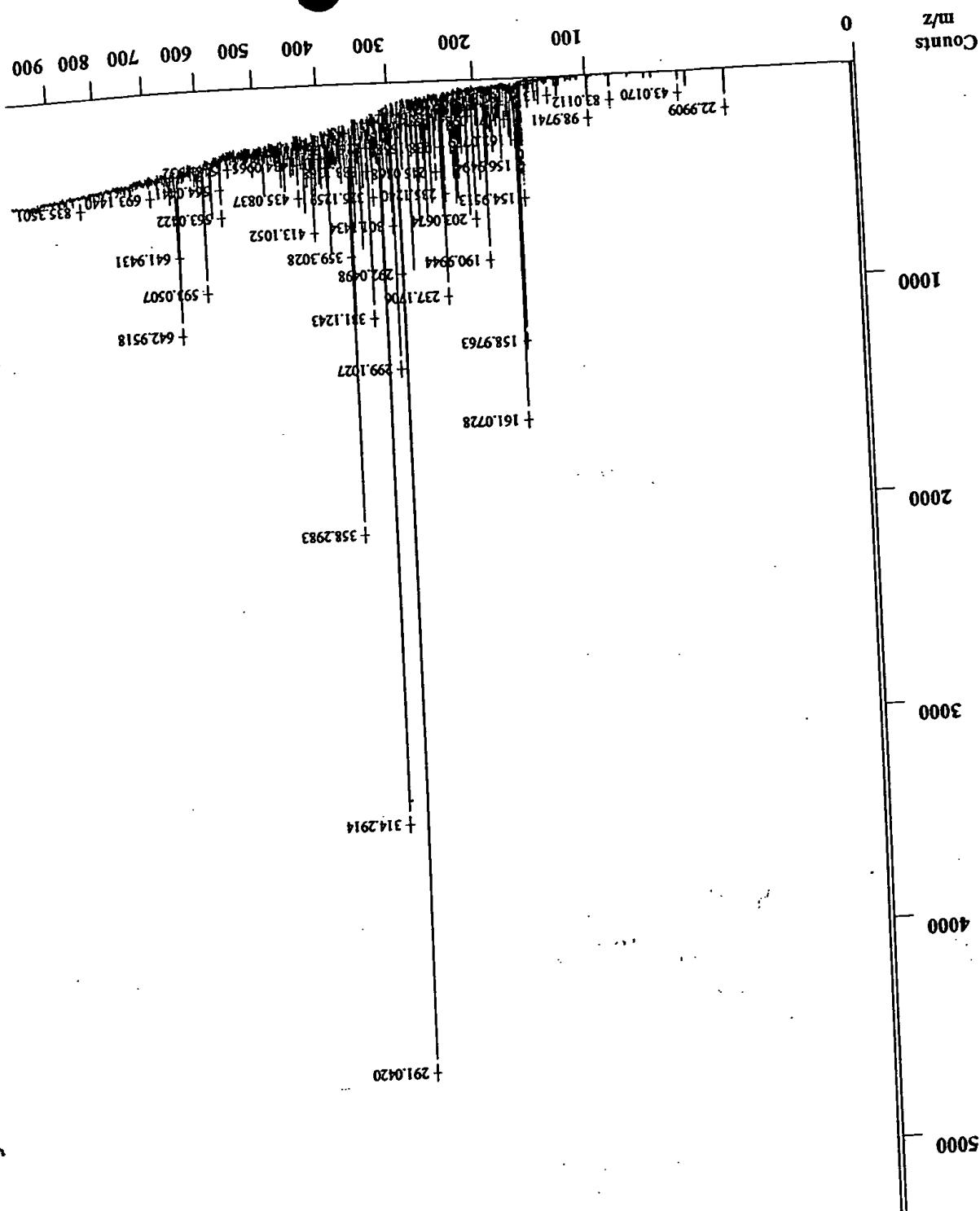
1000 900 800 700 600 500 400 300 200 100







- FIGURE 13 -



Jaguar Summed Mass Spectrum Report

File Name: G:\41.dat
Time Run: 11/13/00 11:01:40 AM
Report Created: 11/13/00 11:03:26 AM

Instrument: JAGUAR

Sample ID: Spec # Range: 2706 - 2778

Method Name: G:\41.dat

Operator: Ionization Mode: ESI - positive ions

TOE214 - ESI/ESI-5000

TO 200 " 5000"

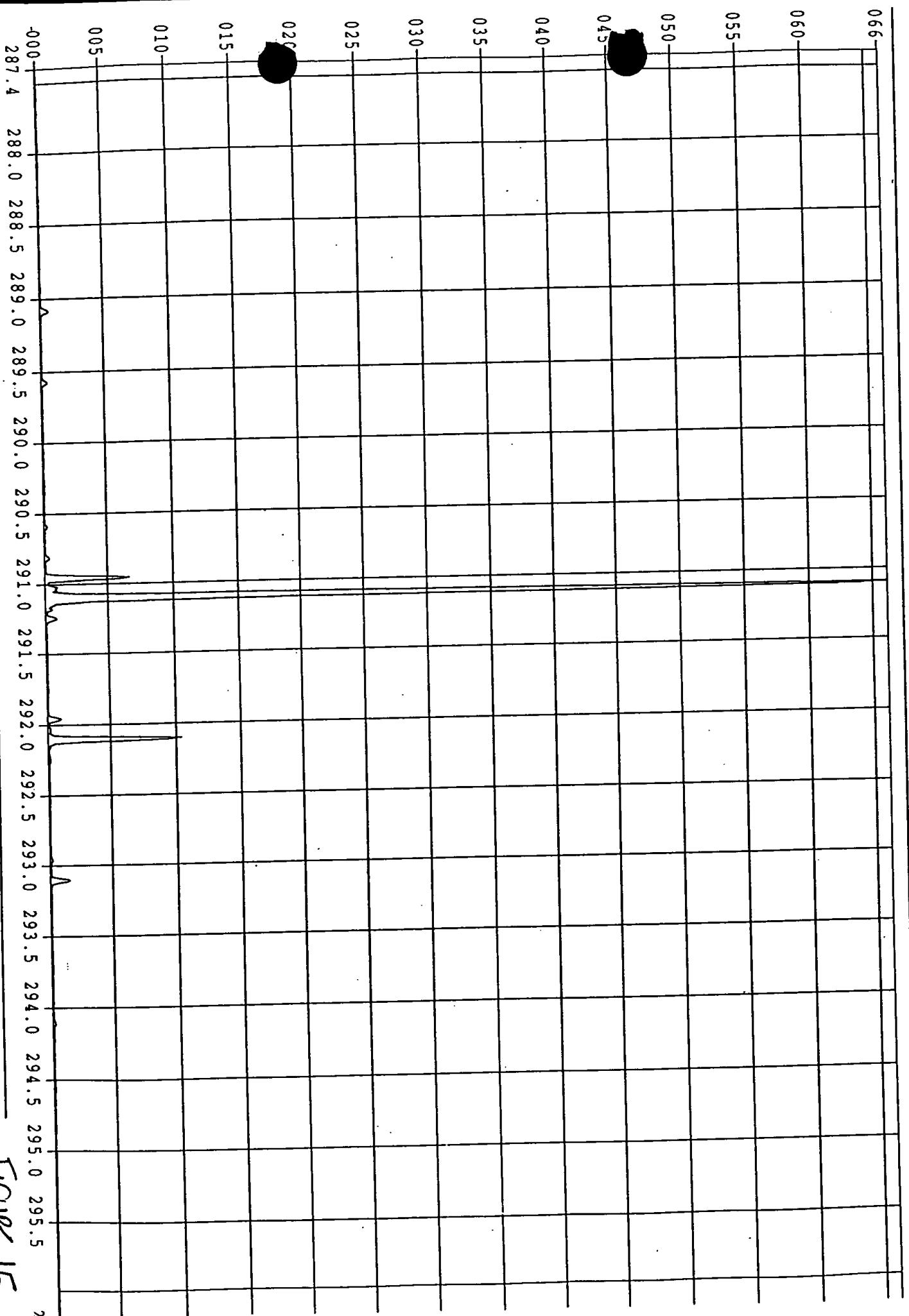


FIGURE 15 -

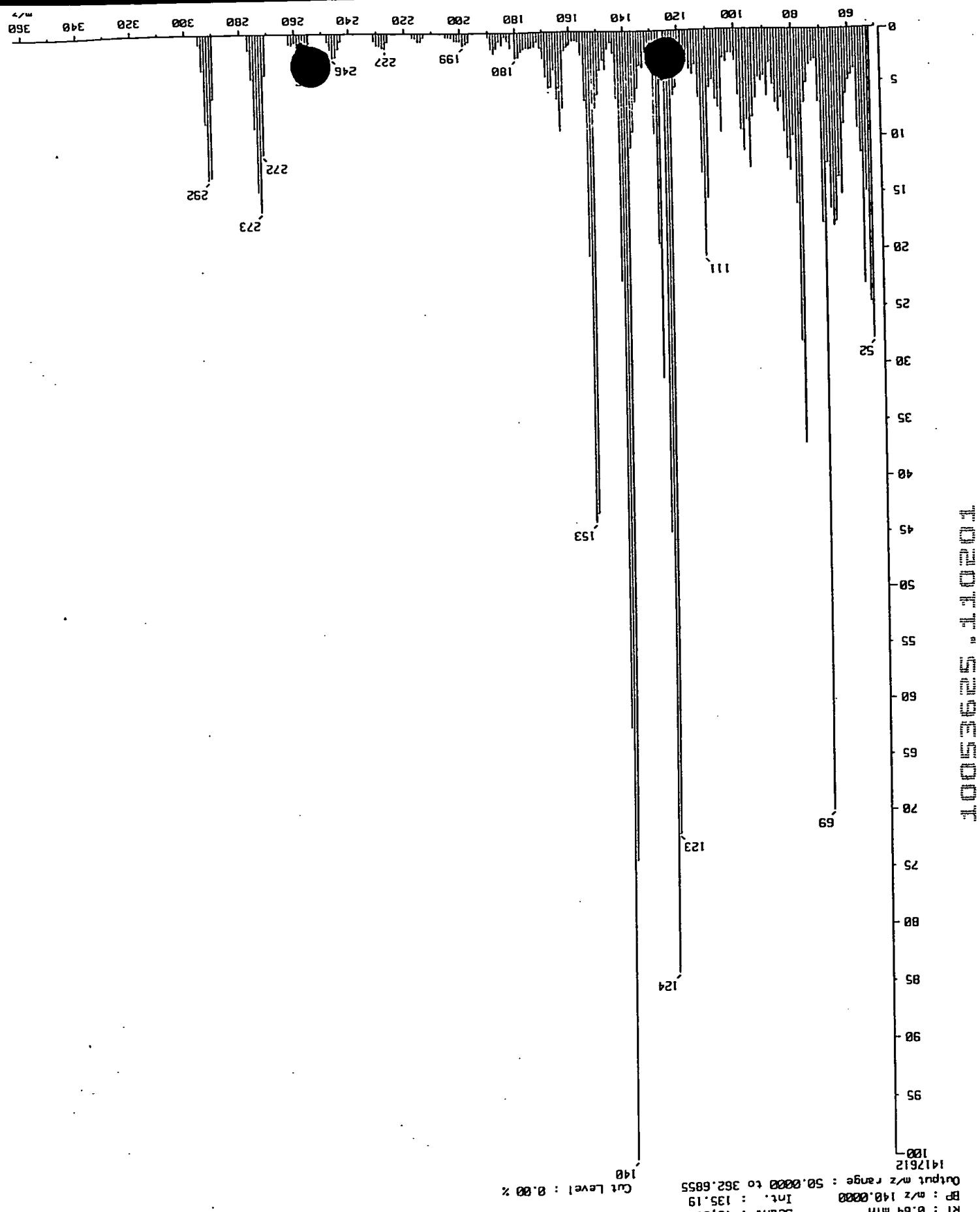


FIGURE 16

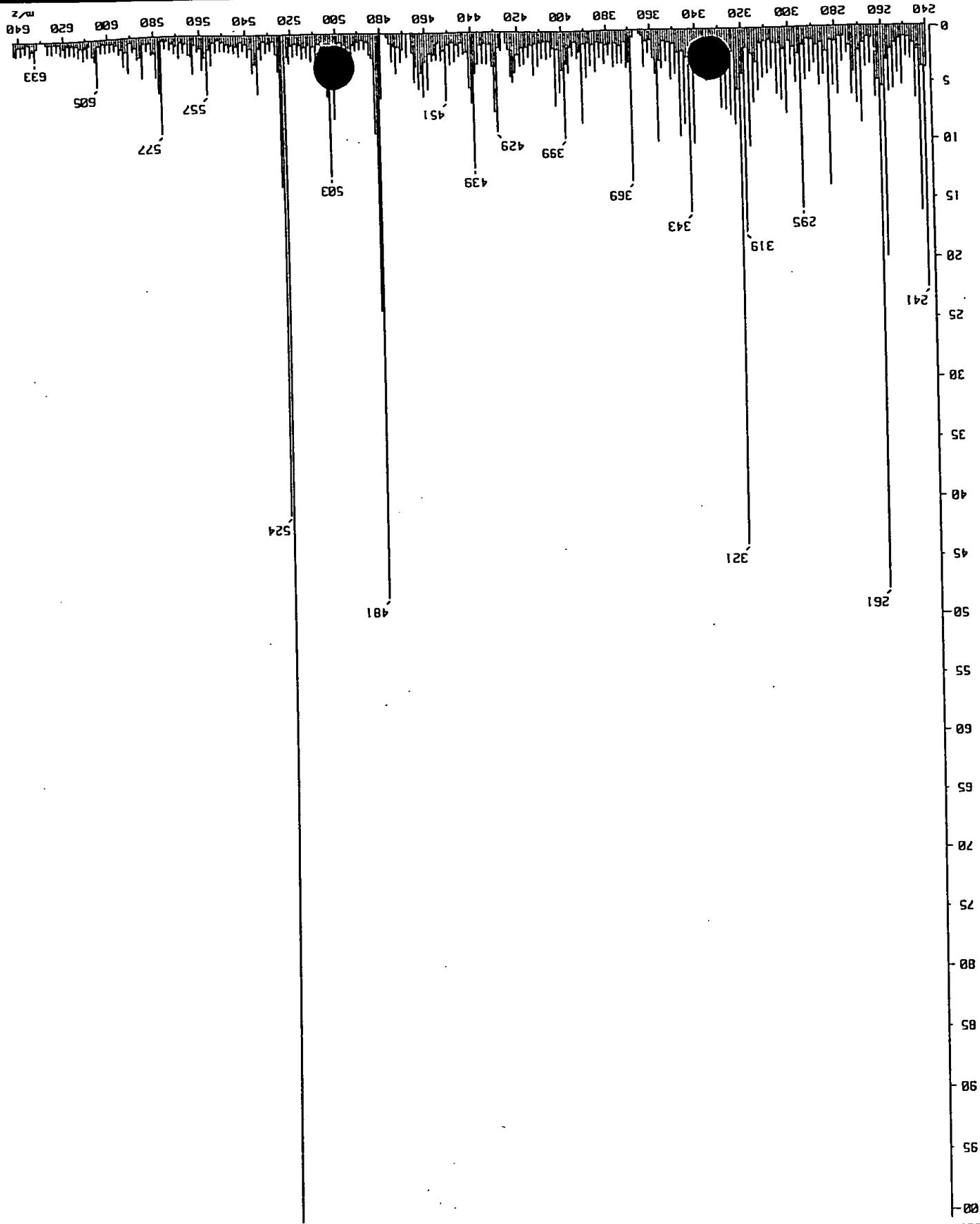
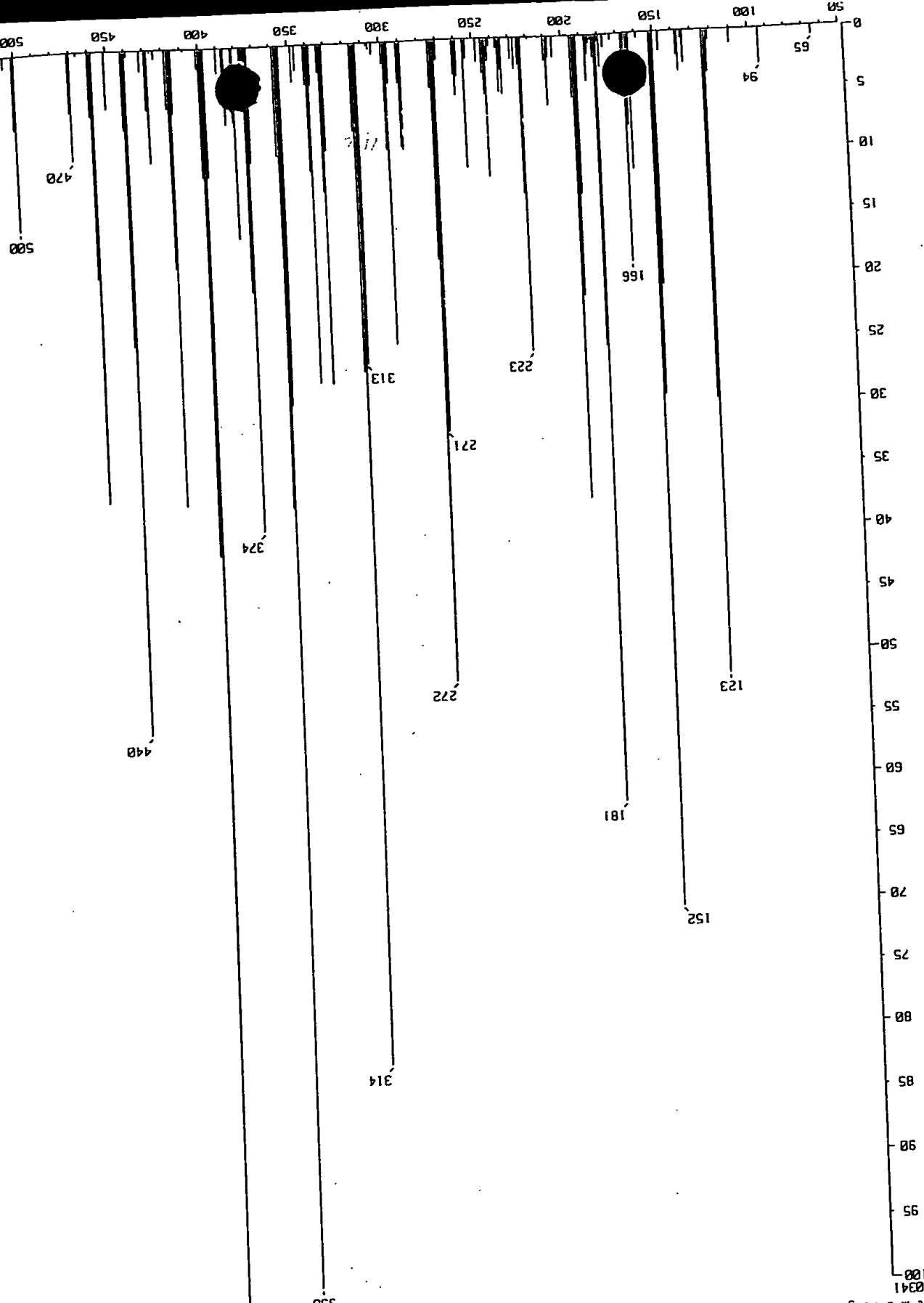


FIGURE 17

FIGURE 18



Mass Spectrum]
Data : BYU 373
Sample: acylated-1
Notes: Inlet: Direct

TOPOTIC SAMPLE

H1 of sample.

Pulse Sequence: 82pul

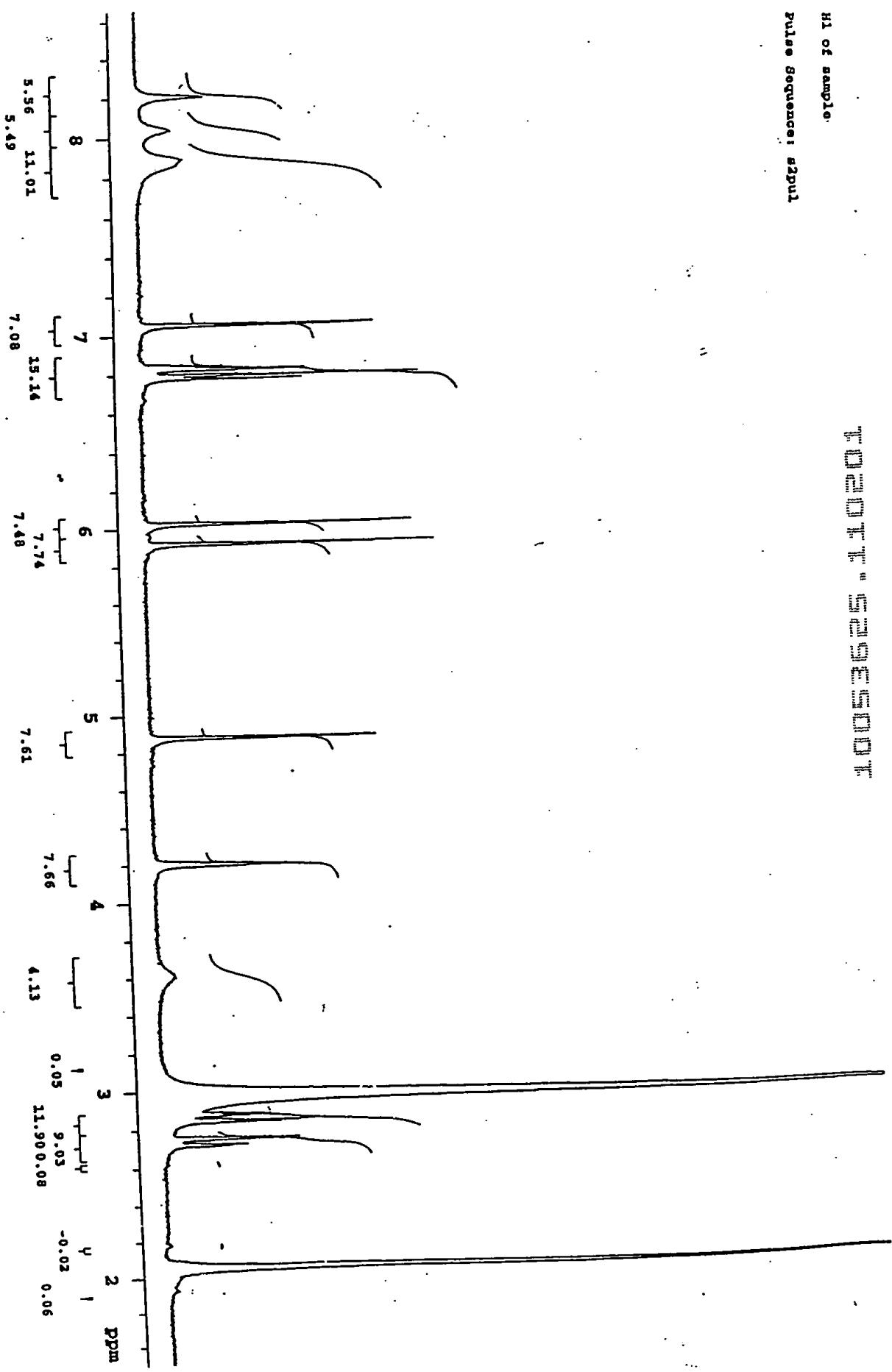


FIGURE 19

¹³C of Sample: In Acetone-d6

TOOTHE 5000

Pulse Sequence: 82pul

306.4

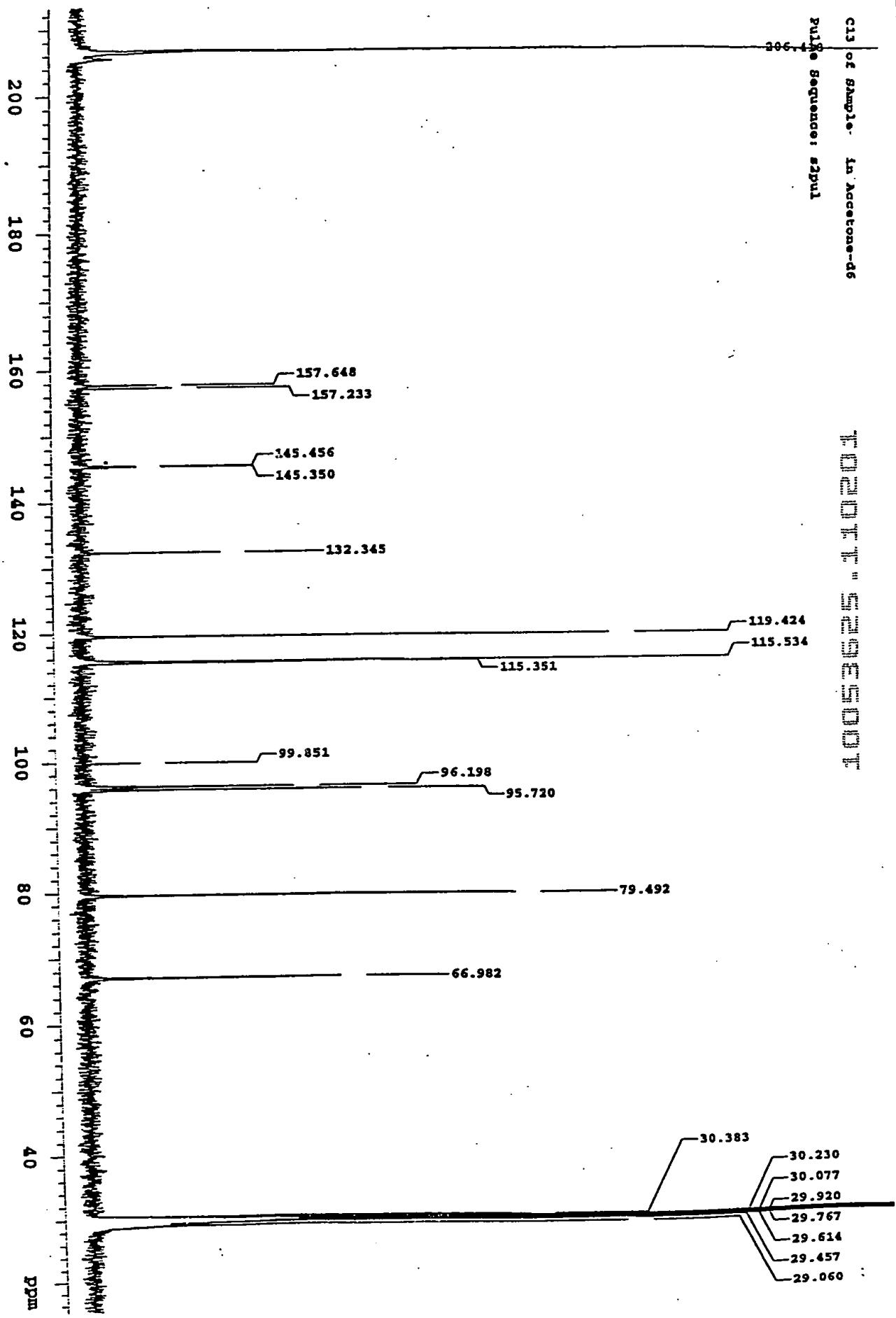
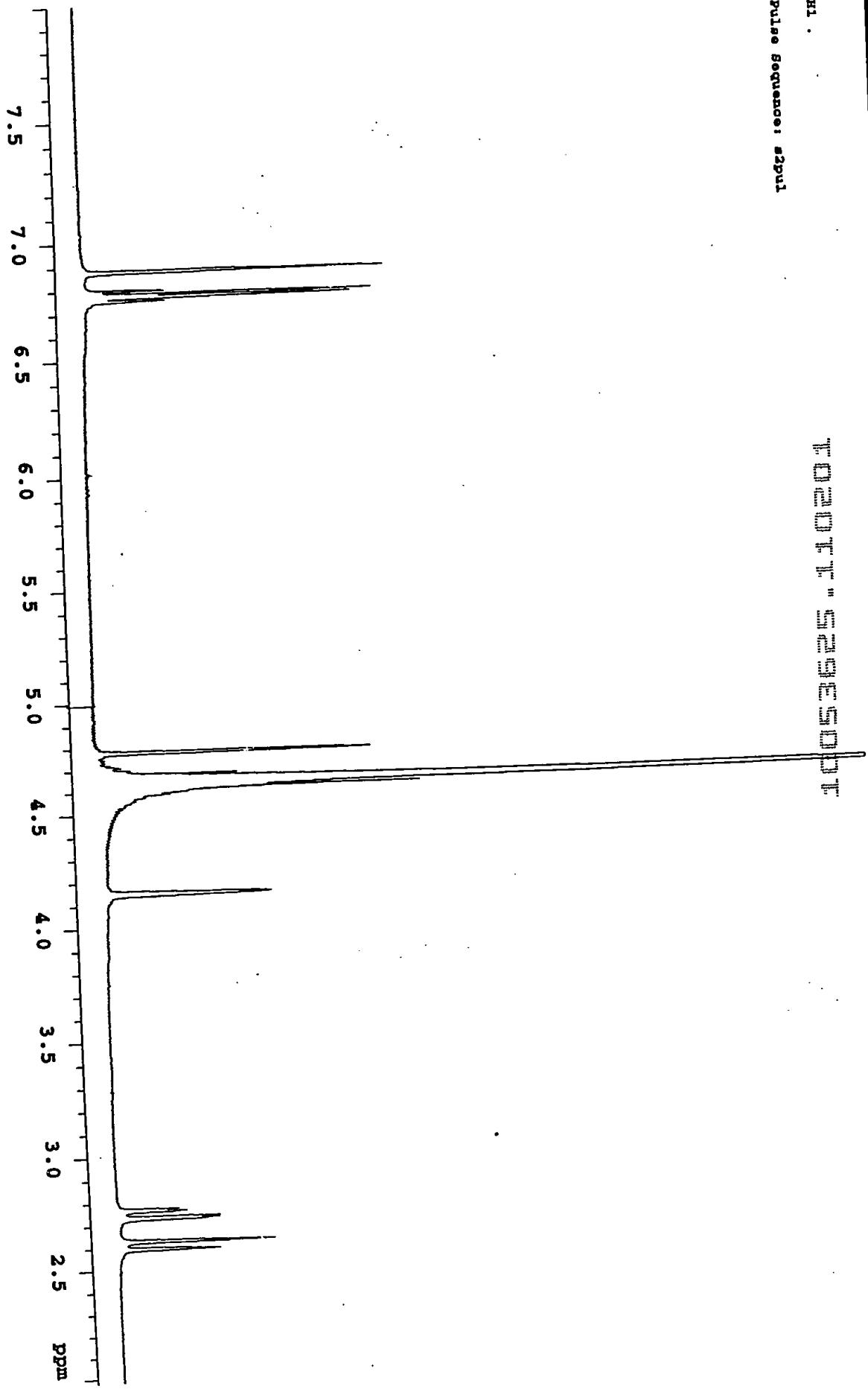


FIGURE 20

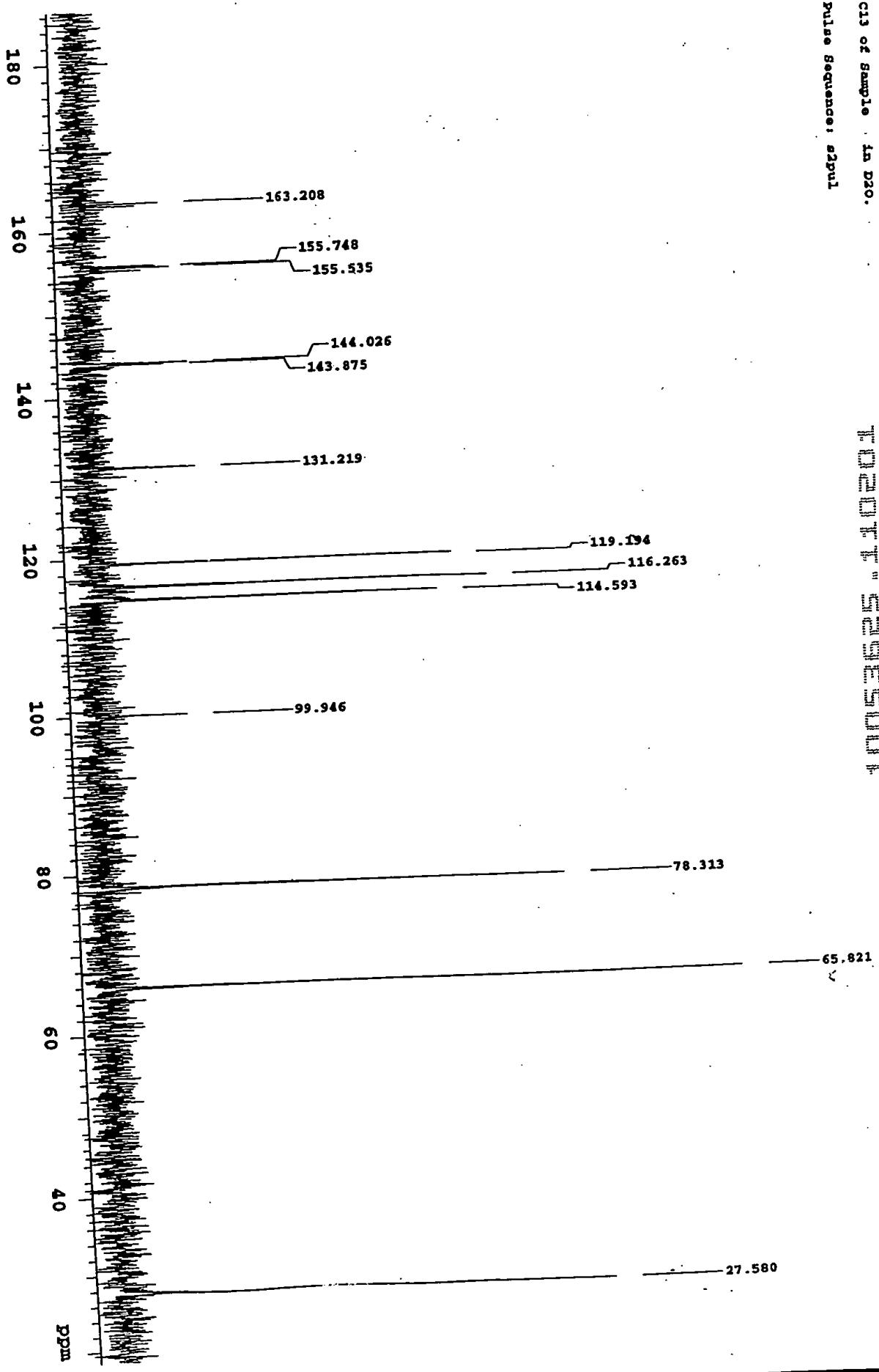
— FIGURE 21 —



C13 of Sample in D2O.

Pulse Sequence: 62w1

TOOT "SINGLET"



— FIGURE 22 —

COSY of Sample...

Pulse Sequence: relayh

Solvent: Acetone

Ambient temperature

UNIV-500 "univ500"

PULSE SEQUENCE: relayh

COSY 90-90

Acc. time 0.303 sec

Width 3817.5 Hz

2D Width 3817.5 Hz

16 repetitions

159 increments

OBSERVE H1, 499.8814428 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 1024 x 1024

Total time 14 min, 12 sec

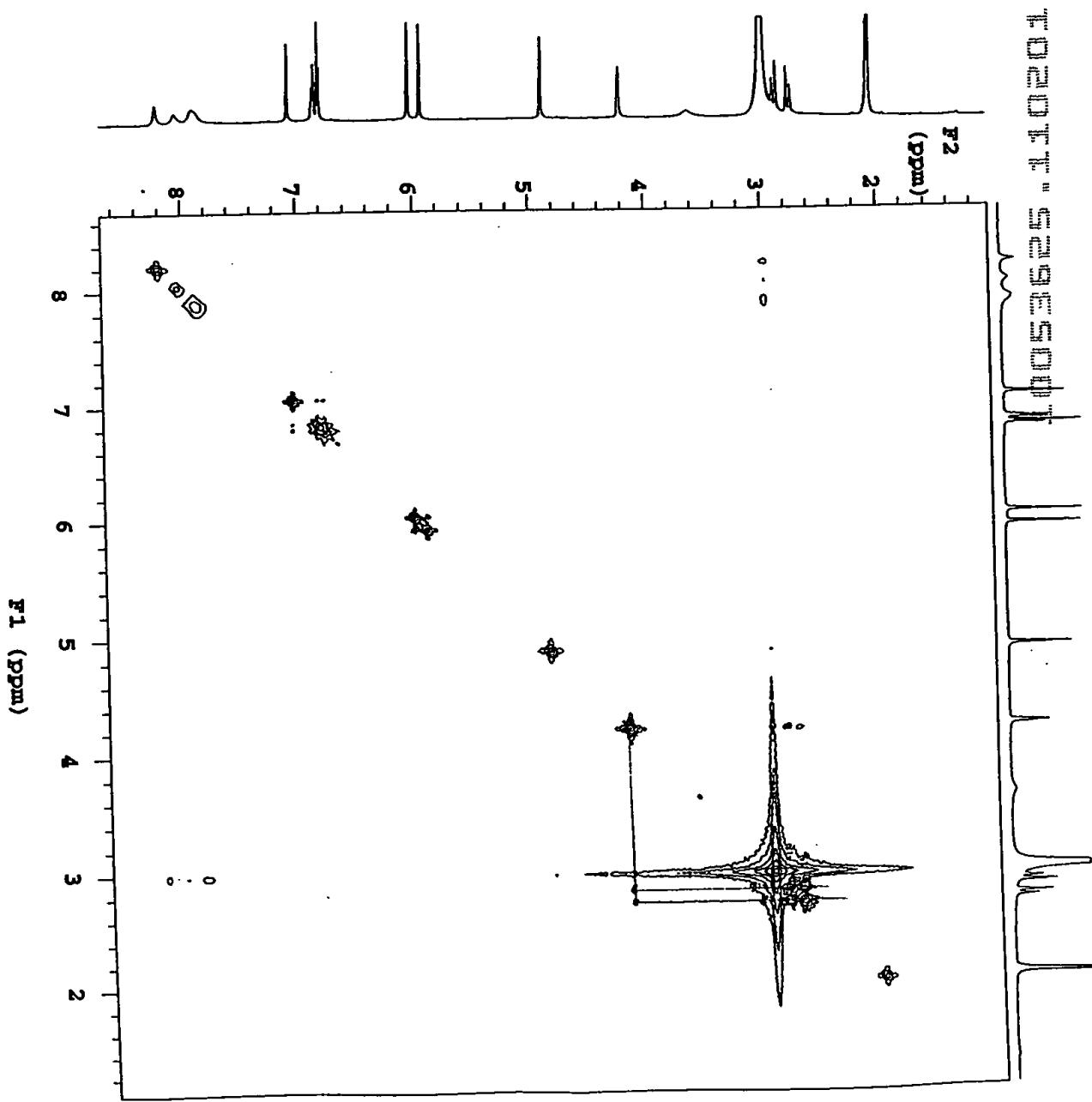


FIGURE 23

COSY

Pulse Sequence: relayb

Solvent: D2O

Ambient temperature

UNIVAC-500 "UNIV500"

PULSE SEQUENCE: relayb

Relax. delay 0.500 sec

COSY 90-90

Acc. time 0.178 sec

Width 2882.3 Hz

2D Width 2882.3 Hz

8 repetitions

120 increments

OBSERVE H1, 499.8801324 MHz

DATA PROCESSING

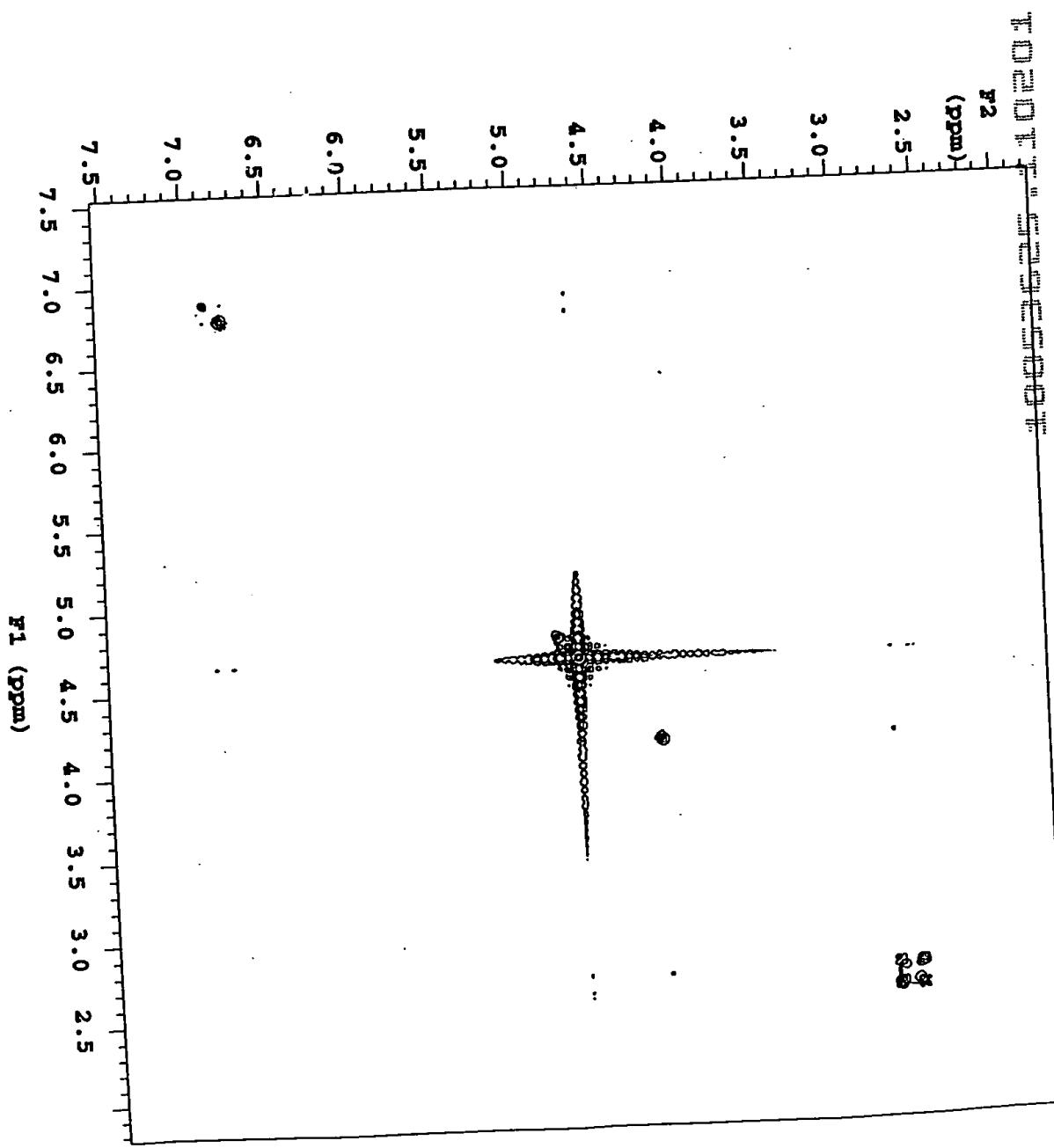
Line broadening 0.1 Hz

T1 DATA PROCESSING

Line broadening 0.3 Hz

FT size 1024 x 1024

Total time 11 min, 23 sec



COSY

Pulse sequence: relayh

Solvent: D2O

Ambient temperature

UNITY-500 "mix500"

PULSE SEQUENCE: relayh

Relax. delay 0.500 sec

COSY 90-90

Acc. time 0.178 sec

Width 2882.3 Hz

2D Width 2882.3 Hz

8 repetitions

120 increments

OBSERVE H1, 499.8801324 MHz

DATA PROCESSING

Line broadening 0.1 Hz

P1 DATA PROCESSING

Line broadening 0.3 Hz

PT size 1024 x 1024

Total time 11 min, 23 sec

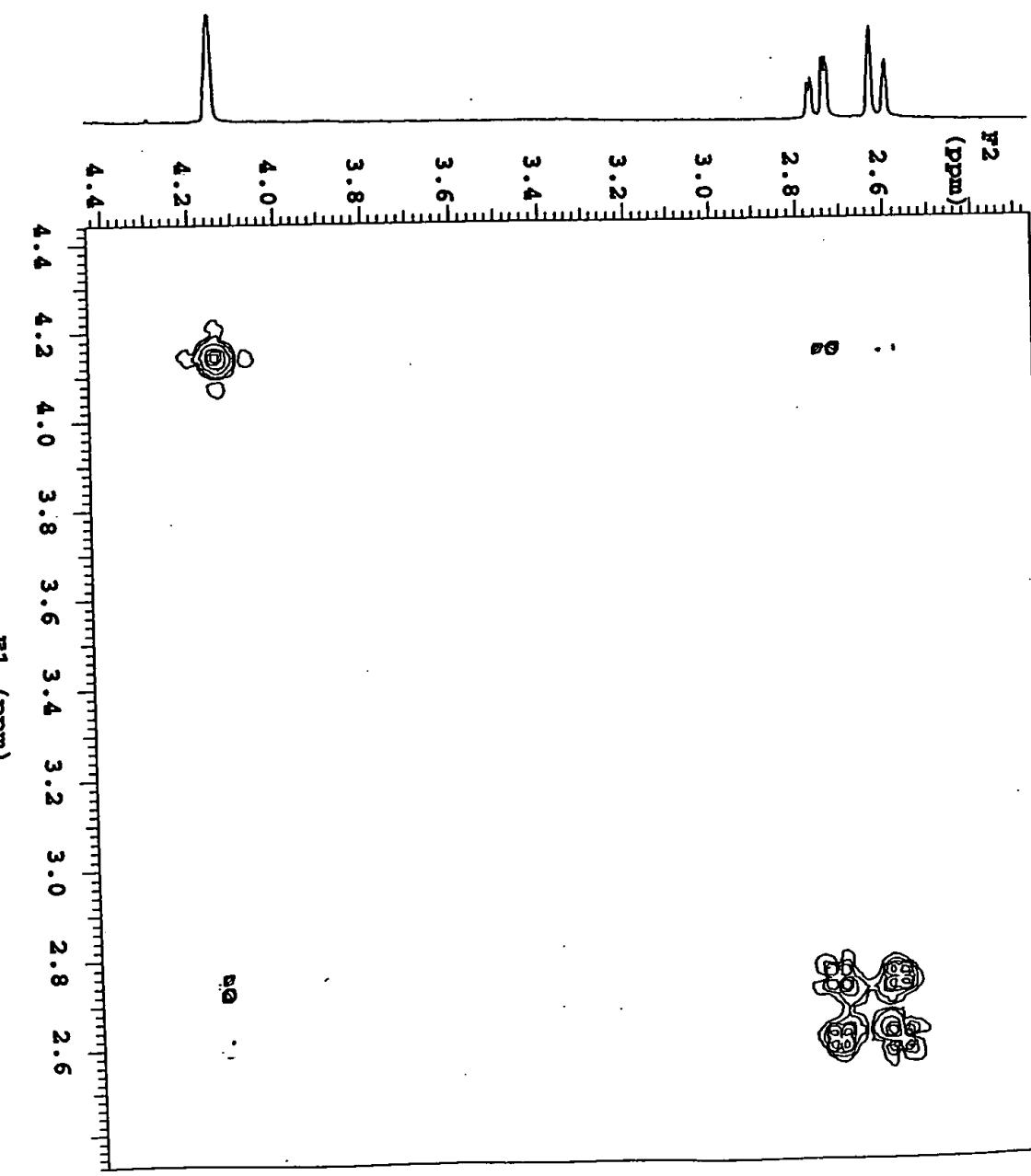


FIGURE 24B

COSY

1020TP "GEME500T

Pulse Sequence: relay2

Solvent: D2O

Ambient temperature

UNIVAC-500 "UNIV500"

PULSE SEQUENCE: relay2

Relax. delay 0.500 sec

COSY 90-90

Acq. time 0.178 sec

Width 2882.3 Hz

2D Width 2882.3 Hz

8 repetitions

120 increments

OBSERVE H1, 499.8801324 MHz

DATA PROCESSING

Line broadening 0.1 Hz

71 DATA PROCESSING

Line broadening 0.3 Hz

FT size 1024 x 1024

Total time 11 min, 23 sec

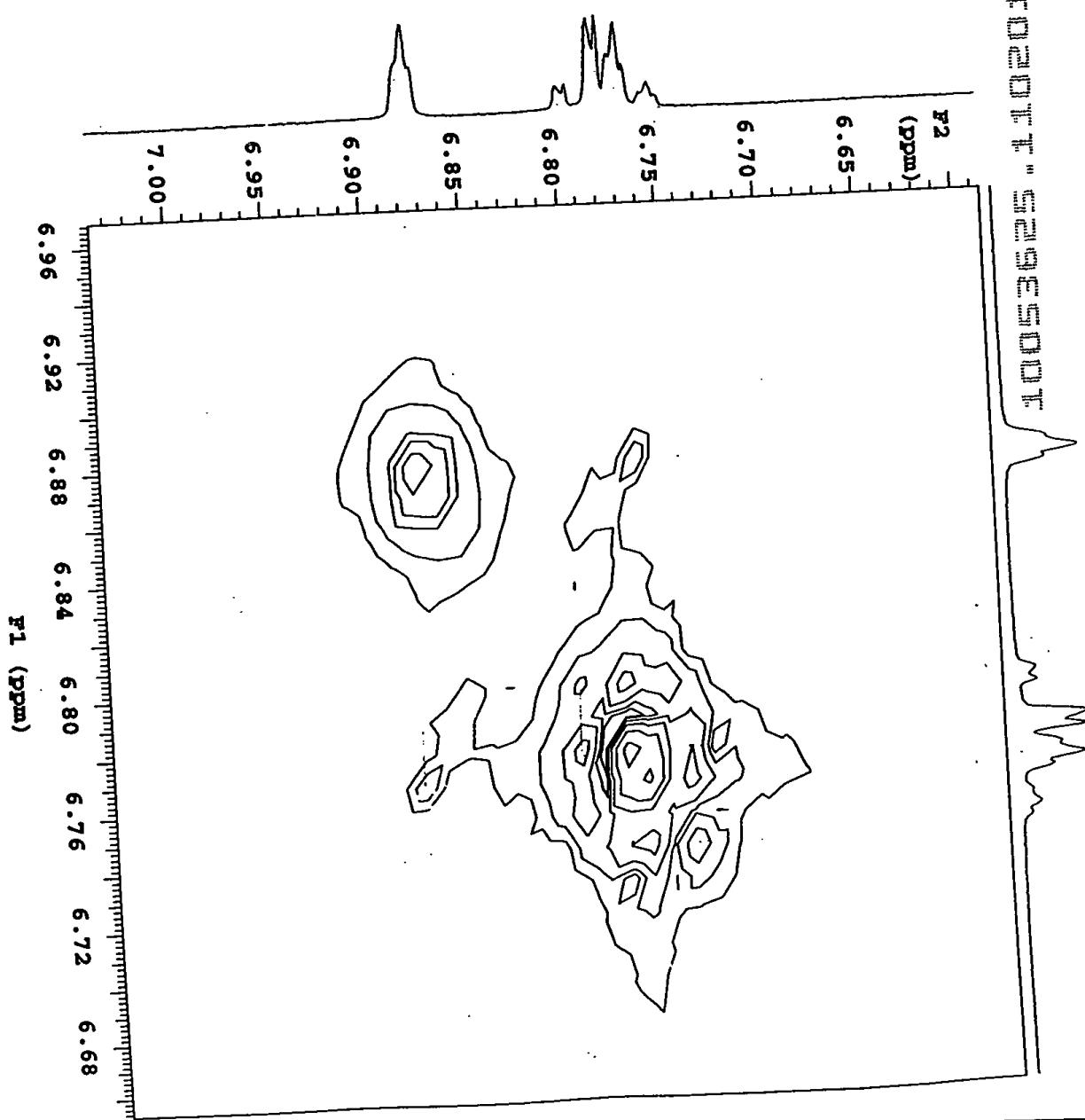


FIGURE 24C

11 of 1 Acetylated in CDCl_3

Pulse Sequence: **2pul**

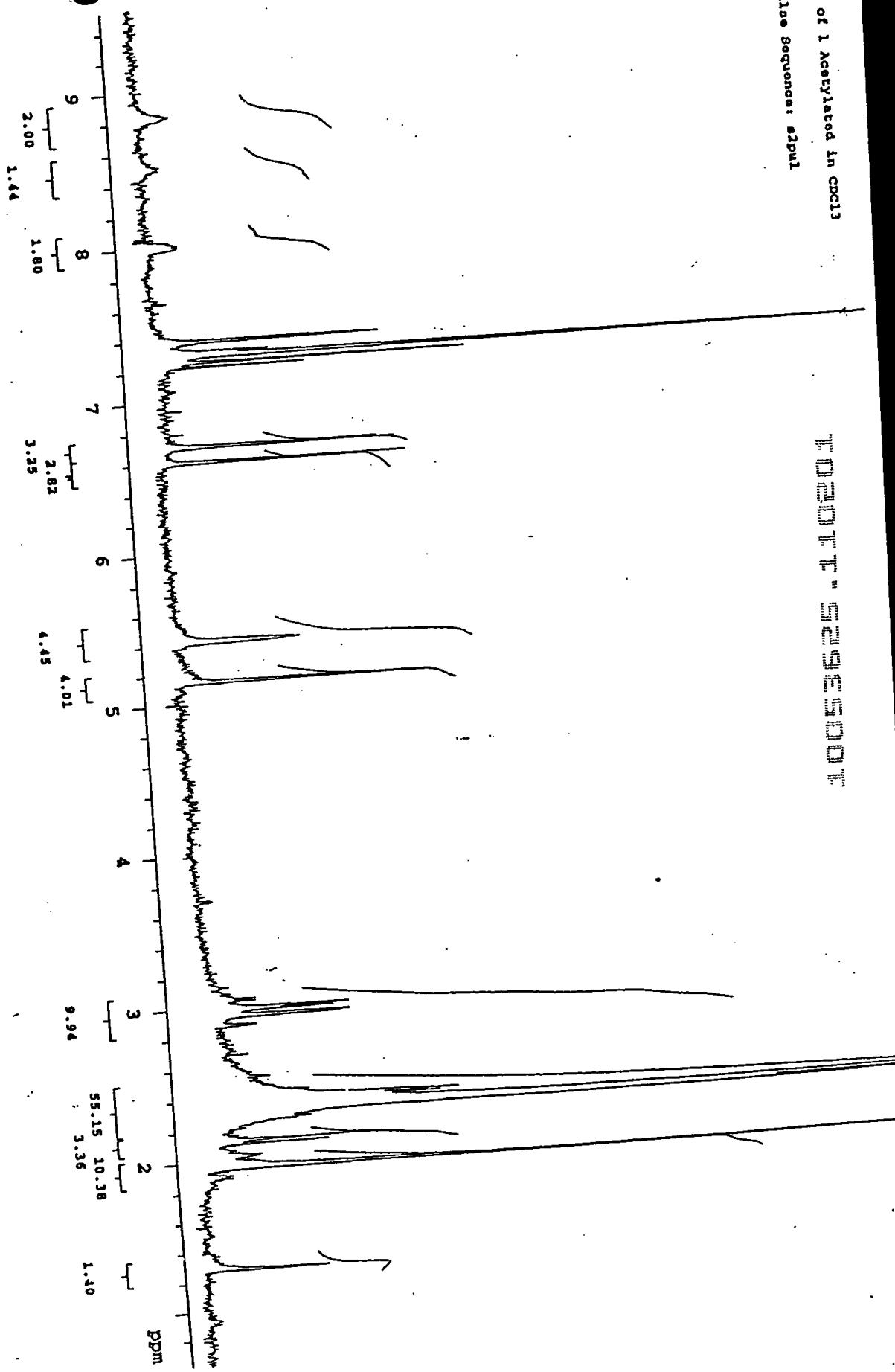


FIGURE 25

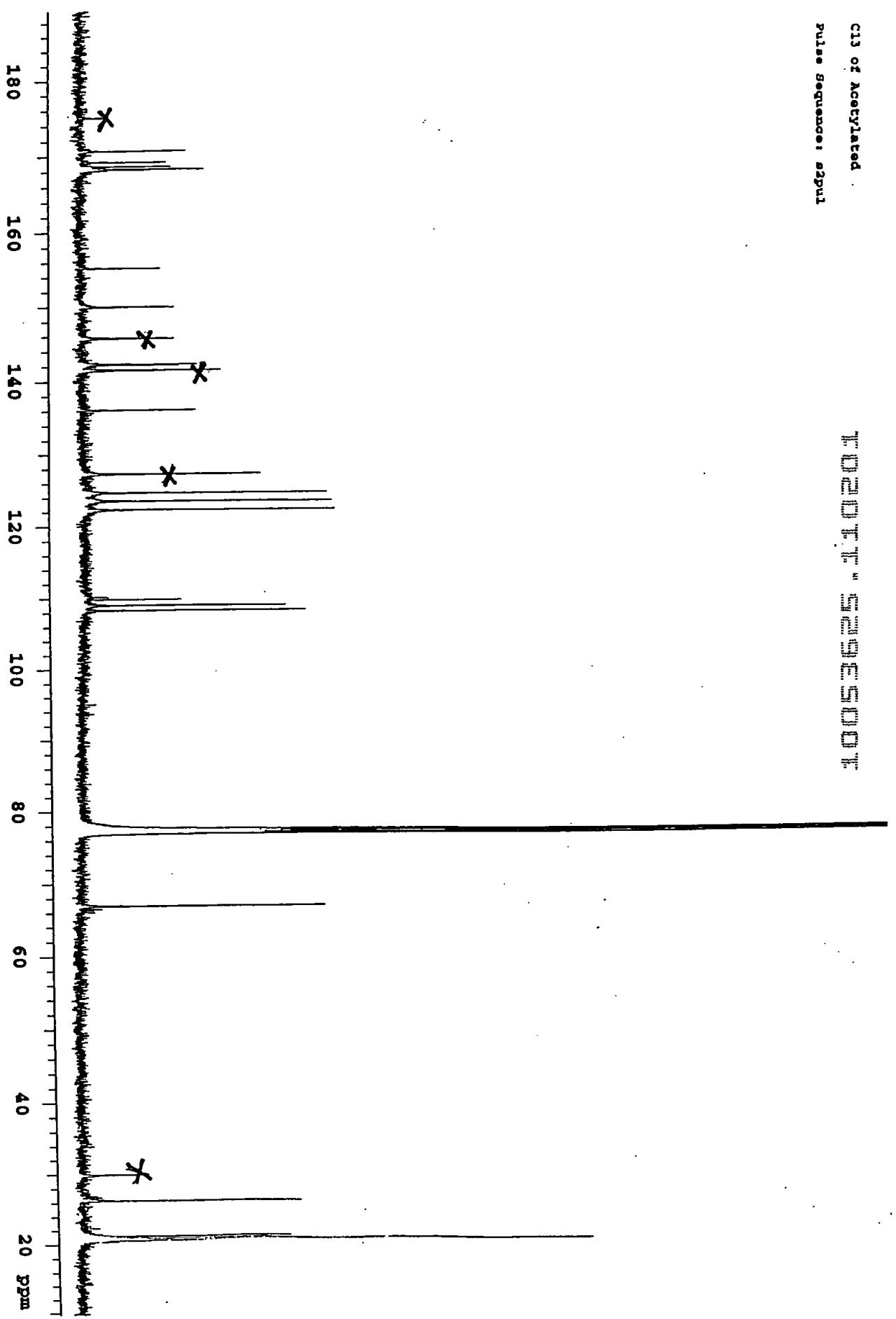


FIGURE 26

COSY

Pulse Sequence: relayb

Solvent: CDCl₃

Temp. 26.0 C / 299.1 K

INOVA-500 "mr500"

Relax. delay 0.500 sec

COSY

90-90

Acq. time 0.128 sec

Width 8000.0 Hz

2D width 8000.0 Hz

16 repetitions

512 increments

OBSERVE H₁ 499.9135718 MHz

DATA PROCESSING

Sine bell 0.064 sec

Y1 DATA PROCESSING

Sine bell 0.032 sec

FT size 2048 x 2048

Total time 1 hr, 31 min, 32 sec

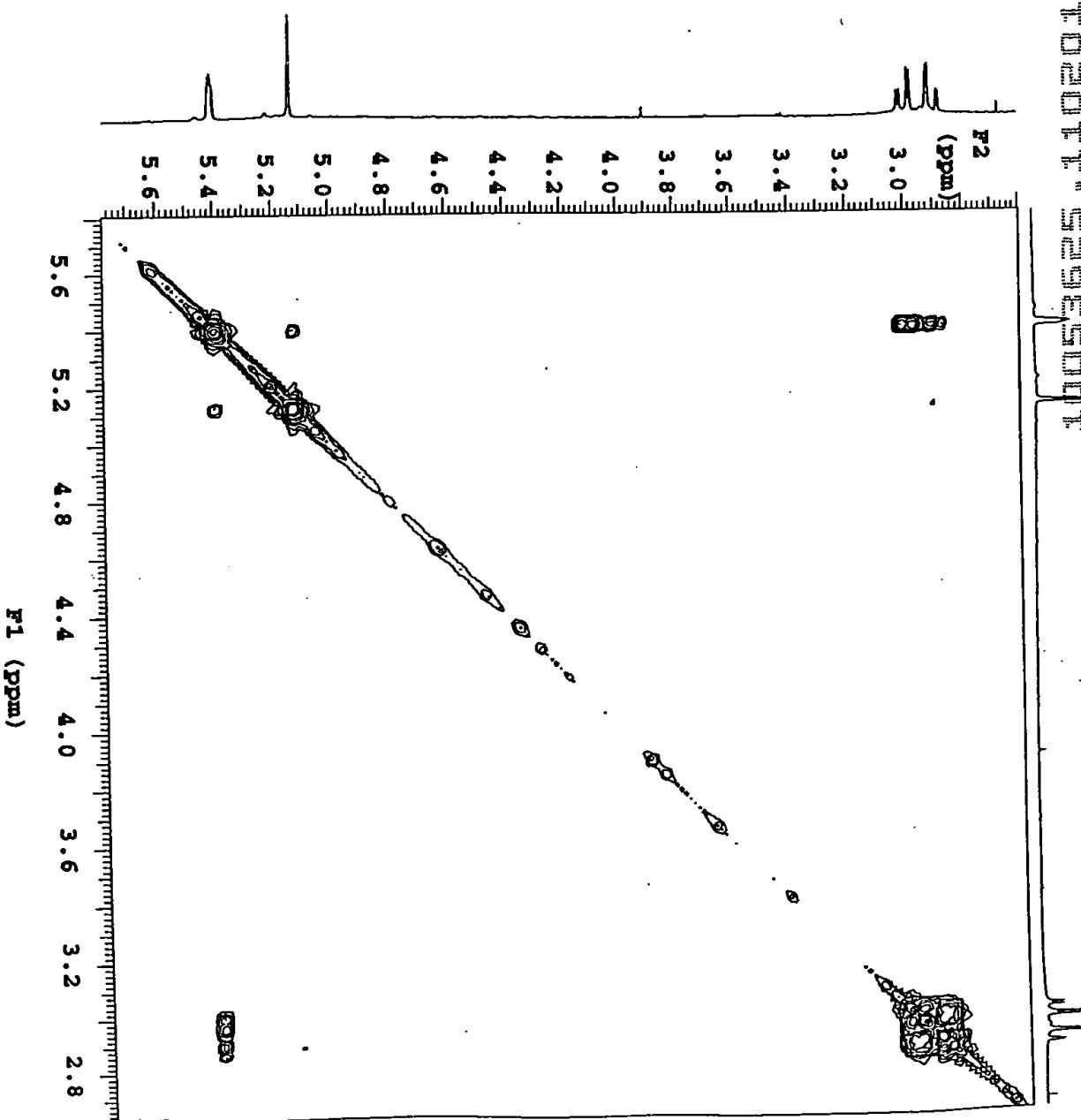


FIGURE 27

RETICOR of Acetylated in CDCl₃.

TOOT - 3292500T

Pulse Sequence: hetcor

Solvent: CDCl₃

Temp. 26.0 C / 299.1 K

User: 1-14-87

INOVA-500 "num500"

Relax. delay 1.000 sec

Acc. time 0.062 sec

Width 25000.0 Hz

2D Width 4614.9 Hz

208 repetitions

256 increments

OBSERVE C13, 125.703376 MHz

DECOUPLE H1, 499.9160715 MHz

Power 38 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

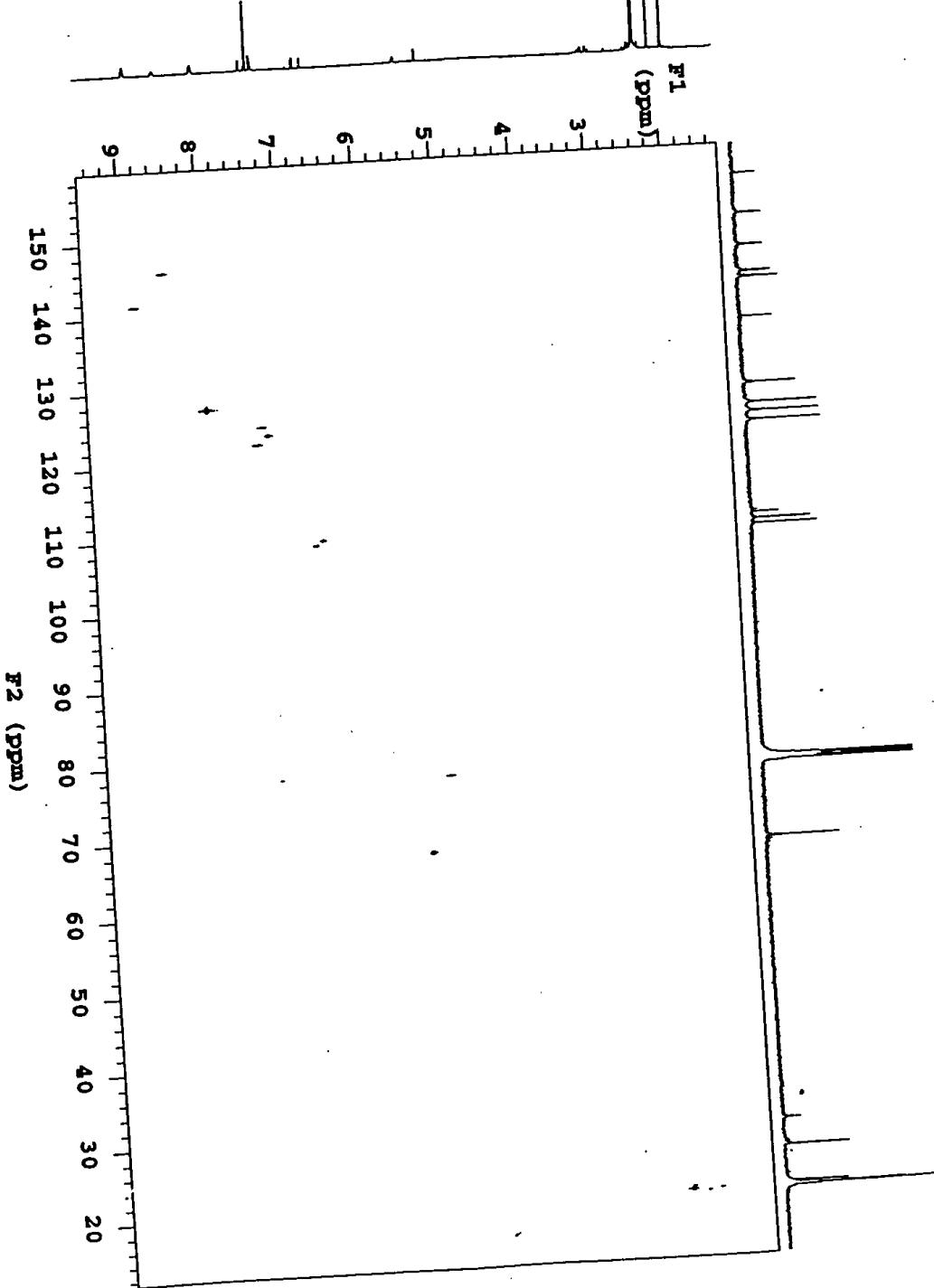
Line broadening 2.0 Hz

F1 DATA PROCESSING

Line broadening 0.3 Hz

PT size 4096 x 1024

Total time 16 hr, 46 min, 21 sec



— FIGURE 28A —

NETCOR of Acetylated in CDCl₃.

Pulse Sequence: netcor

Solvent: CDCl₃

Temp. 26.0 C / 299.1 K

User: 1-14-87

INOVA-500 "nmr500"

Relax. delay 1.000 sec

Acq. time 0.082 sec

Width 25000.0 Hz

2D Width 4614.9 Hz

208 repetitions

256 increments

OBSERVE C13, 125.7033376 MHz

DECOUPLE H1, 499.9160715 MHz

Power 38 dB

on during acquisition

off during delay

WALTZ-16 modulated

DATA PROCESSING

Line broadening 2.0 Hz

1D FID PROCESSING

Line broadening 0.3 Hz

72 slices 4096 x 1024

Total time 16 hr, 46 min, 31 sec

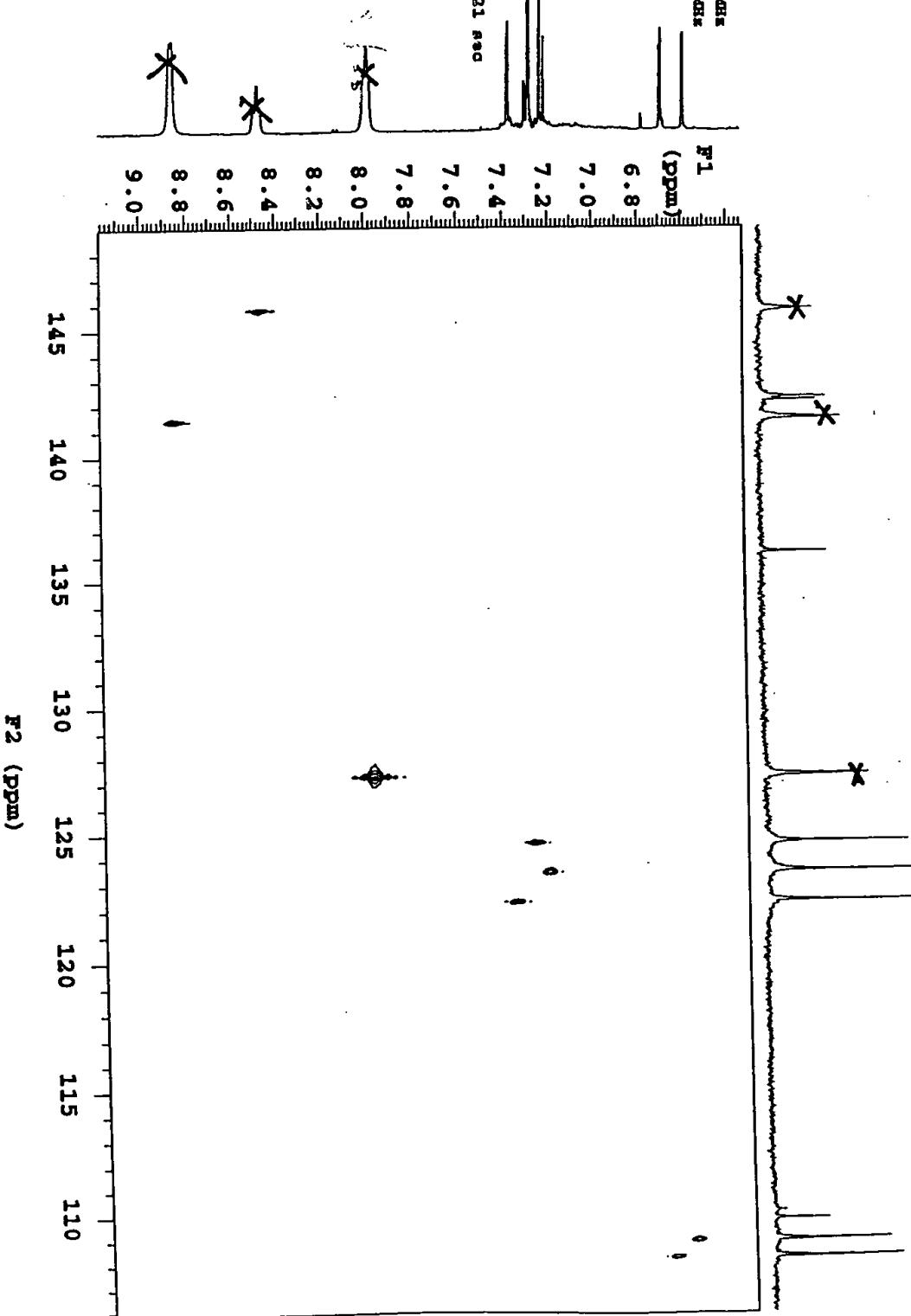


FIGURE 28B

HETCOR of Acetylated in CDCl_3 .

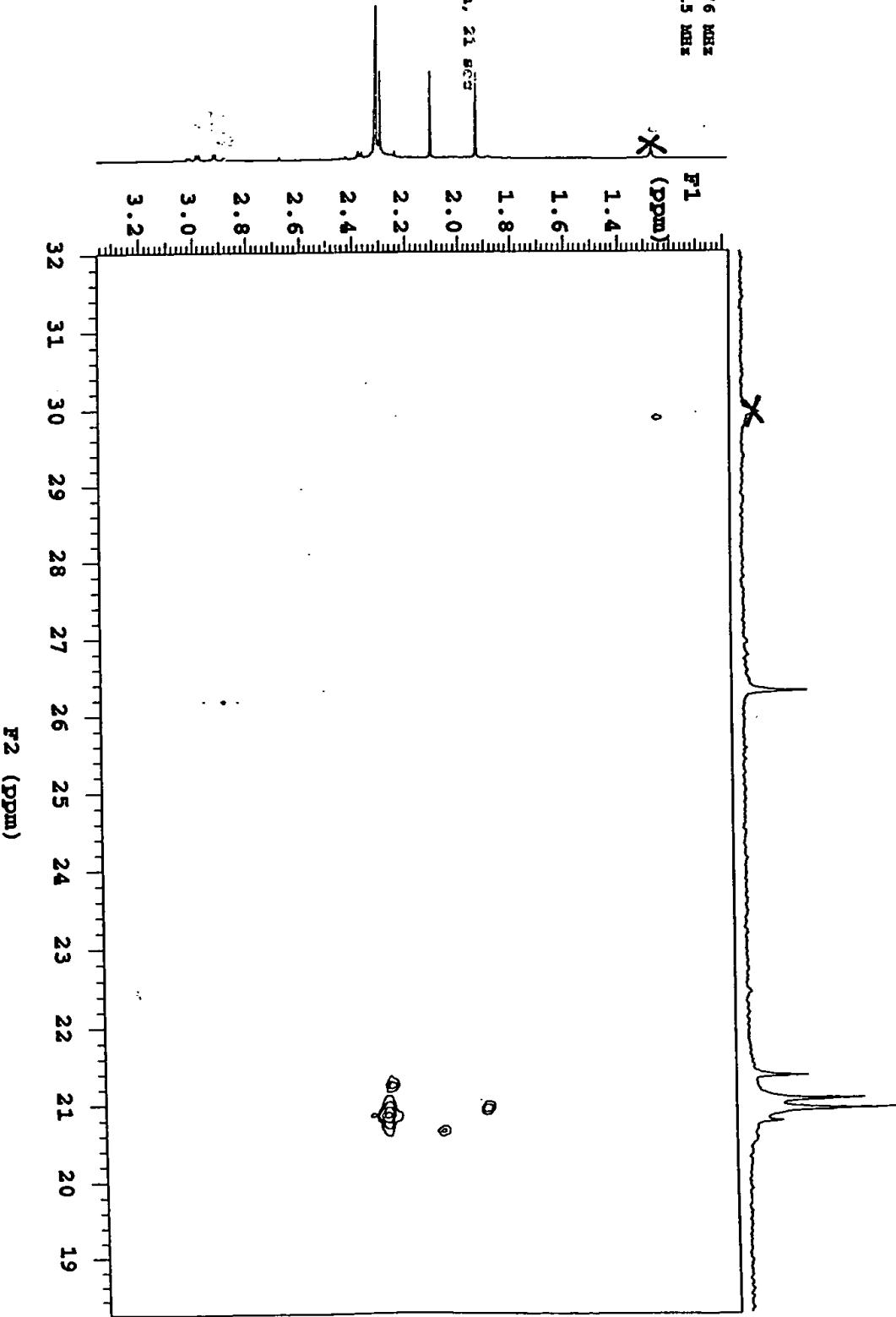
Pulse Sequence: hetcor
Solvent: CDCl_3
Temp. 26.0 C / 299.1 K
User: 1-14-87
INOVA-500 "mmx500"

X = Pyridinium acetate

Relax. delay 1.000 sec
Acq. time 0.082 sec
Width 25000.0 Hz
2D Width 46144.9 Hz
208 repetitions
256 increments

OBSERVE C13, 125.7033376 MHz
DECOUPLE H1, 499.9160715 MHz

Power 30 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
PPM 4996 x 1024
Total time 16 hr, 46 min, 21 sec



— FIGURE 28C —

FIDDLER™ SPECTROSCOPIC

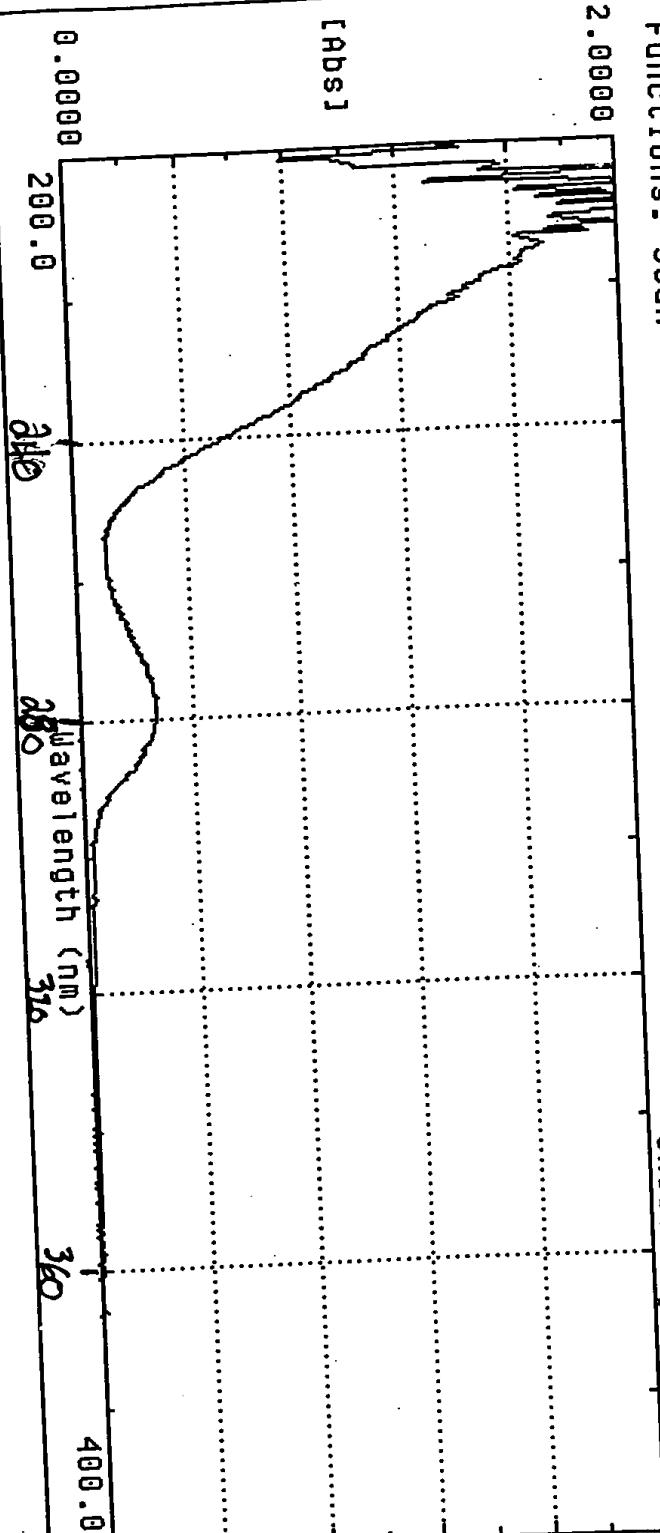
HELP

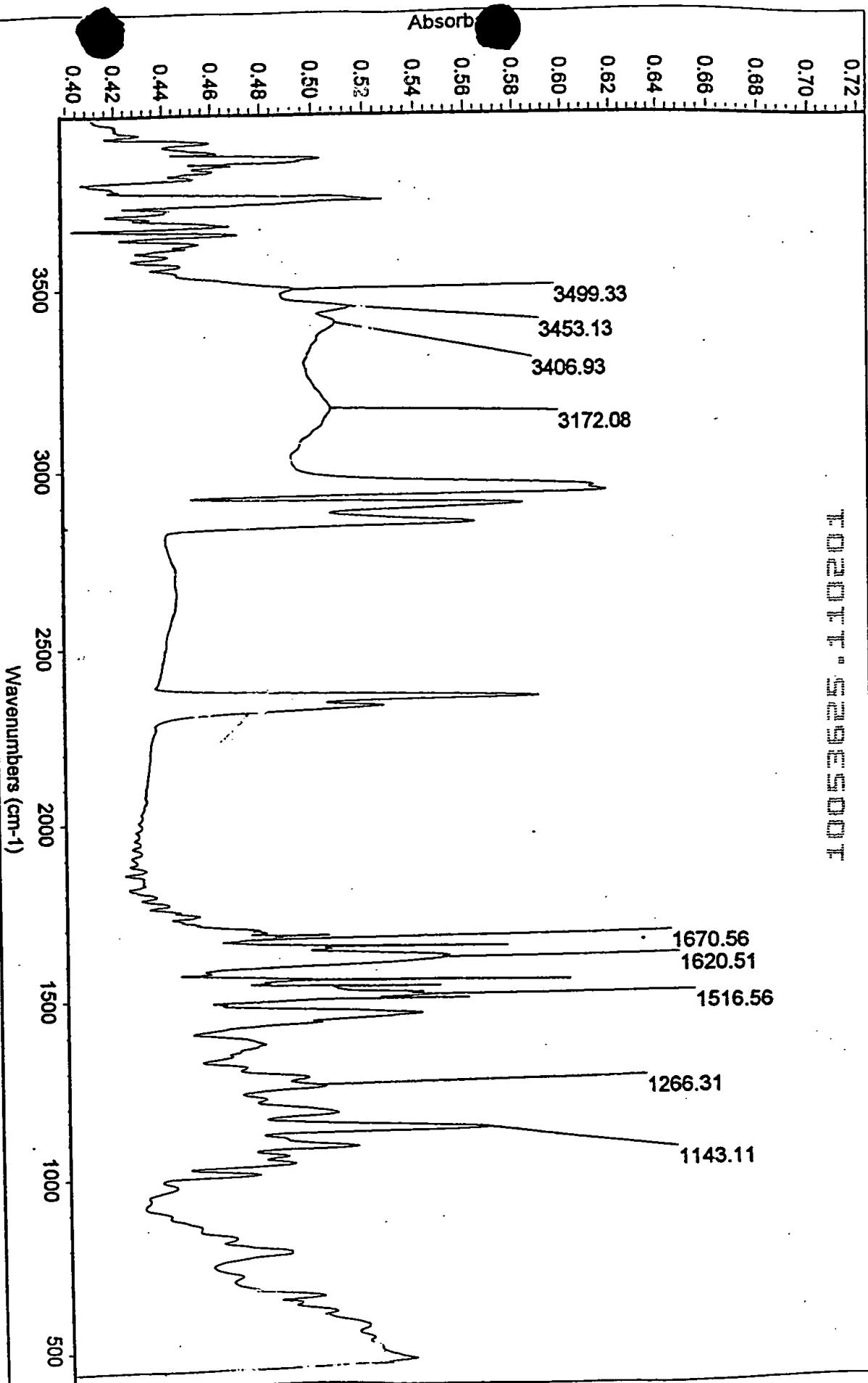
Wavelength Scan Tabulate +-* /Scans Scatter NetA Method SaveClear Print Quit
 ReadSamples

Scan directory: VIEW Autoprint: [No] Method name: A:\DEFAULT
 Start w1: 200 nm Autosave: [No] Autosave name: LA:\VSCANS
 End w1: 400 nm Scans per sample: 1 Sampling device: None
 Overlay scans: [No] Scan speed: 600 nm/min

A:\WORK_005 (600)

Zoom ZoomOut Trace Function Autoscale Annotate Print
 Functions: Scan Smoothing: None





**Subtraction Result: proteolach, sampleL

Scans: 32

Resolution: 4.000

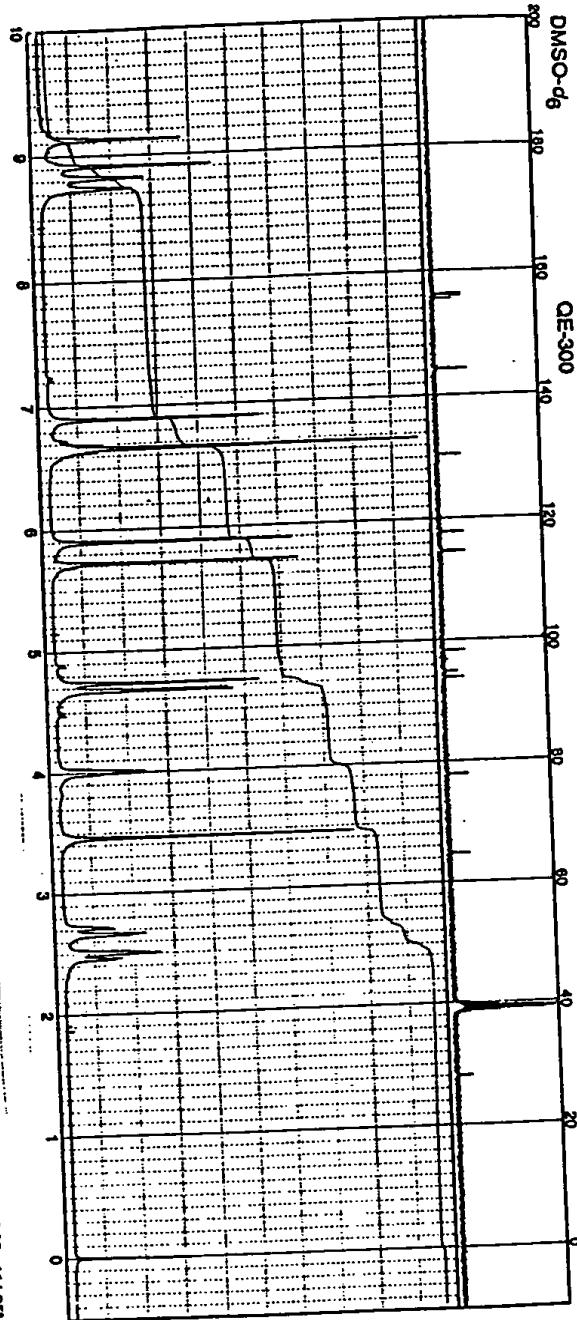
— FIGURE 30 —

Aromatic Alcohols and Phenols

Aldrich 85,523-5 CAS[480-46-0] $\text{C}_15\text{H}_{14}\text{O}_6$ mp 280.27
 (-)-Epicatechin, 97% FW 280.27
 mp 240°C.d.

156.31 130.42 94.93°
 156.01 117.78° 83.91°
 155.67 114.59° 77.98°
 144.29 114.58° 64.75°
 144.23 98.31 28.07

A



Aldrich 86,181-2
 (±)-Catechin hydrate, 98%
 $\text{C}_15\text{H}_{14}\text{O}_6$
 FW 280.28
 mp 210°C.d.

156.25 114.35°
 155.97 94.90°
 155.15 94.97°
 144.65 93.70°
 130.43 80.83°
 118.24° 68.17°
 114.92° 27.70

C

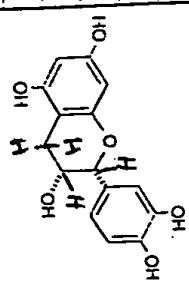
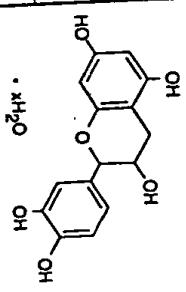


FIGURE 31

A

TOOT T T" SCALES 0.00 T

IR III

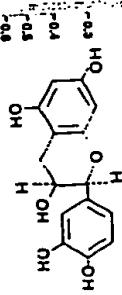
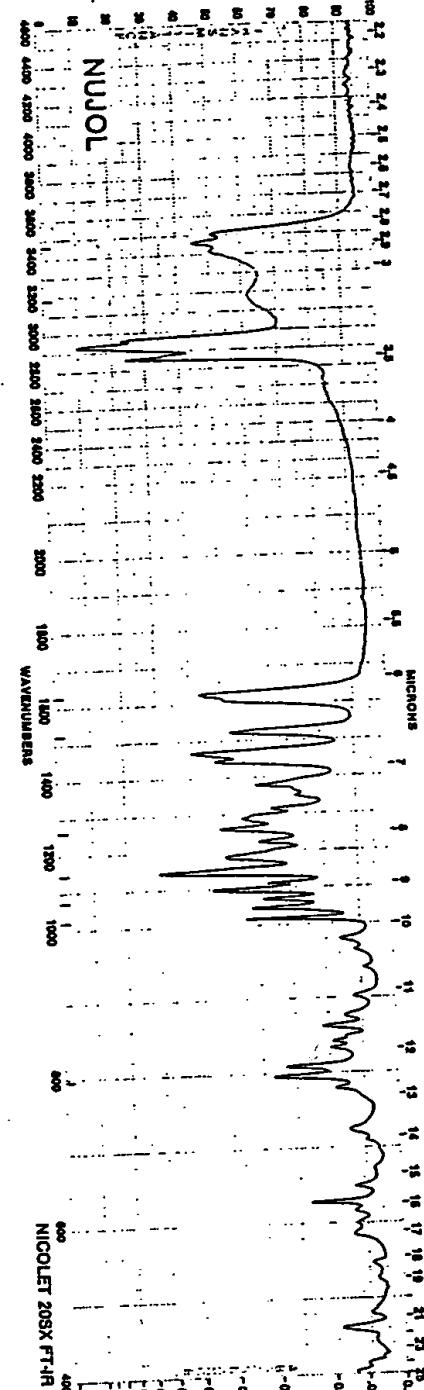
3455.2 1521.4 1069.5
3176.0 1281.0 1018.8
1626.3 1144.3 784.7

D

85523-5 CAS [480-46-0]
l-Epicatechin

FW 290.27
mp 240°C (dec.)

IR III, 673E



22402-2 CAS [154-23-4]
(+)-Catechin hydrate

FW 290.28

IR III, 703G
Merck 10,1833

3411.2 1514.7 1144.3
3134.9 1293.2 1020.6
1609.9 1236.4 832.3

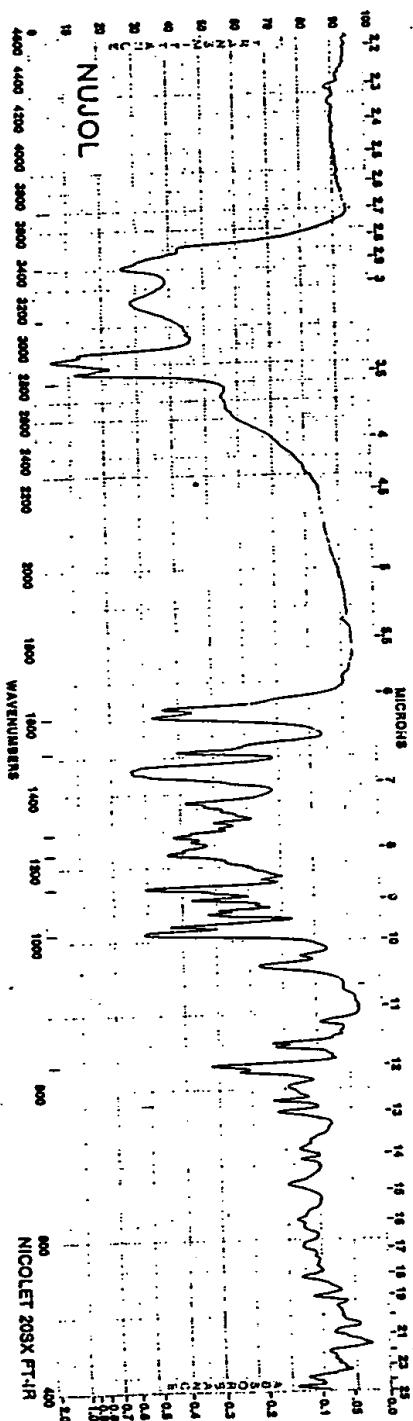


FIGURE 3B

FIGURE 33

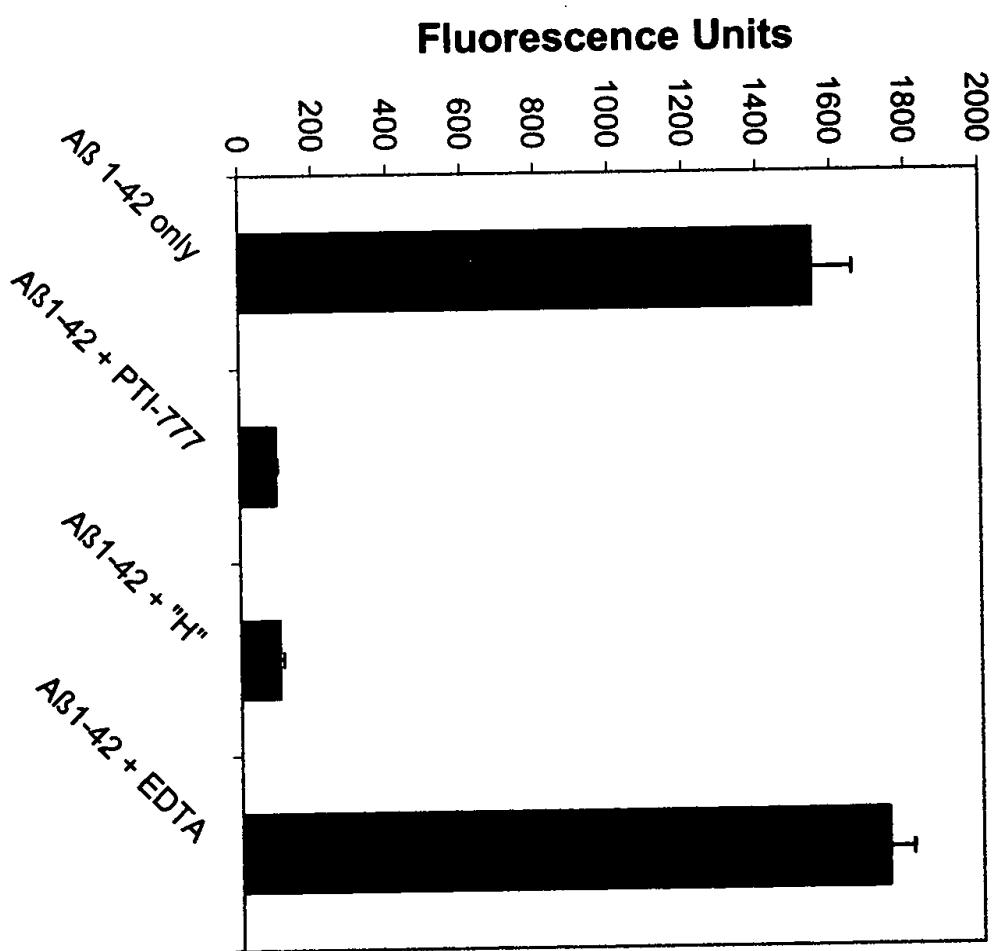


FIGURE 34

